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THE INSTITUTE OF PETROLEUM

AN Ordinary General Meeting of the Institute of Petroleum was held at 61 New Cavendish Street, London, W.1, on 2 September 1959, the Chair being taken by C. Chilvers, a Vice-President of the Institute.

The General Secretary read the minutes of the previous meeting, which were confirmed and signed as a correct record. He also announced the names of members elected since the previous meeting.

The Chairman introduced the author and the following paper was then presented in summary.

THEORY OF ACID TREATMENT OF LIMESTONE FORMATIONS*

By G. ROWAN†

SUMMARY

This paper is devoted to the study of the problem of the increase in permeability of porous limestone formations due to acid treatment and proposes a theory of the process under certain simplifying assumptions. These assumptions are similar to those adopted for the prediction of permeability from pore size distribution data.

Previous work on the theory of acid treatment by French authors resulted in a formula which has been applied to the interpretation of laboratory tests to determine the susceptibility of limestones to acid treatment. This theory presupposes a uniformity of effective pore structure and consequently is of limited validity.

In view of the discrepancies existing between laboratory investigations and theoretical predictions, a somewhat different approach has been made to the problem. This approach is based on non-uniformity of pore structure, in recognition of the fact that, in general, the effective pore structure may be expected to be composed of a distribution of pore sizes. Calculations based on the proposed theory appear to be in better accord with the results of laboratory investigations.

The results of this study demonstrate the dominant effect of the larger pores and the rapid increase in permeability obtainable when a distribution of pore size is present. It appears inevitable that the major portion of the acid ultimately passes through only the large pores and, in view of their great contribution to the overall permeability, any attempt to block them off, in the hopes of attaining more uniform acidizing, must result in a lower permeability increase and a less economical employment of the acid.

INTRODUCTION

BEFORE embarking upon the detailed study of any particular facet, it is proposed to review, briefly, the wider aspects and implications of the problem of acid treatment of limestone formations, in so far as they affect oil production.

Essentially, the method consists of selecting and isolating a producing formation, or a portion thereof, and injecting hydrochloric acid of some particular concentration, with the object of increasing the permeability in an annular region surrounding the well. The ultimate effect is an increase in production, similar to that which would be obtained, for example, by a considerable increase in borehole diameter. The aim of acid treatment, however, is not the increase in borehole diameter, for this would be a most uneconomical procedure, besides which further production problems would doubtless be introduced. On the contrary, the object is to prevent such increases in order to obtain the maximum benefit within the formation. Increased productivity, therefore, results from a redistribution in the pressure pattern around the well, brought about by readjustment of formation permeability.

Consider now the mechanism of the process and try to follow the manner in which such changes are produced. Upon injection into the formation, the acid will displace, to a certain extent, the fluids already present in the pore space. Unless the acid is retarded, chemical reaction between the limestone and the acid will commence as soon as contact is established. Thus, on passing into the formation, the concentration of the acid will decrease progressively, owing to continuous chemical reaction along the flow path. The distance to which a given volume of acid will penetrate into a formation, before becoming spent, will depend on the following factors: the permeability and porosity of the formation, the nature of the fluids present, the initial concentration of the acid, the type of limestone, the reaction rate of the acid, and the formation temperature and pressure. No doubt additional factors might intervene, but the above mentioned are sufficient for the present discussion.

How, then, do these factors influence the process? To answer this question let us consider a small volume of acid passing through an elementary volume of the formation. The local velocity of the acid will be governed by the usual laws of fluid motion in a porous medium, and will therefore depend on the local values

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† The British Petroleum Co. Ltd.

of permeability, effective porosity, and pressure gradient. The residence time of the acid in the element of rock will be a function of its velocity and hence a function of these variables. During its time of residence within the element, the acid will react with the limestone, causing an increase in porosity and hence permeability. The extent of the increase will depend upon the amount of pore space contacted during the residence time, upon the reaction rate of the acid with the particular type of rock, its concentration, and the temperature and pressure in the formation. To a greater or lesser degree, most of these variables will depend upon the nature and quantity of other fluids present at this time.

It will be appreciated, therefore, that an adequate study of the problem of acid treatment would be a very difficult undertaking. One of the major complicating factors, and possibly the one about which the greatest uncertainty will remain, is the effect of fluids other than acid in the formation. Some simplification of the problem results if the assumption is made that these fluids are completely swept from the formation by the advancing acid front. The problem then reduces to the study of the permeability increase of an element of rock, in which the acid is able to contact the whole of the effective pore space.

Although not explicitly stated, it will be obvious that the increase in permeability in a rock element will depend upon its distance from the well bore, *i.e.* upon the distance through which the acid has travelled, with consequent decrease in concentration. Also it will obviously depend upon the amount of acid that has passed through the element prior to the instant under consideration. Hence a more general study of the problem would have as its objective the investigation of the areal changes in permeability in terms of the volume and rate of acid injection. It is proposed to deal with this problem at some future date.

Apart from the primary purpose of acid treatment as a means of stimulating well productivity, there are attendant effects which have an important bearing on other problems. One such problem might be the interpretation of pressure build-up records, taken after such a treatment. It is known that formation damage is reflected in the shape of the pressure build-up curve. Acid treatment, or formation improvement, must surely also have an effect, but this phenomenon has as yet not been clearly formulated.

The question might be raised as to the reasons motivating a detailed study of this problem. The process has been applied successfully in the field for many years with tangible results, and a considerable amount of operating experience has been amassed. The answer is simply that at present we have very little knowledge of the mechanism by means of which productivity is increased. Our methods are largely empirical and rely mainly on the experience of the operator in similar fields. Without any theoretical background on which to base calculations, the evaluation of the effect of an acid treatment is difficult. For example, it is not inconceivable that in the treatment of a tight limestone formation the injection pressures might reach a point at which fracturing might occur at some section. This would result in large permeability increases which might have the disproportionately favourable interpretation of being due to the effect of the acid treatment alone.

In the review of the general problem of acid treatment, an elementary volume of rock was postulated. As in all flow problems, it is necessary to study what happens in an element, or differential volume, in order that the equations describing the process as a whole may be formulated. The theory contained in this paper is concerned with this particular aspect. It is a first step in the wider study of the problem, a differential formulation perhaps. Such a study is necessary, because without such knowledge we cannot proceed to the next stage.

This method of formulation has the advantage that it corresponds to tests in the laboratory, in which small core plugs are acidized to determine the increases in permeability attained in small samples of the formation.

At this juncture, previous work on this particular aspect of the problem should be mentioned. The important and successful contributions to the field technique have already been noted, as has the fact that, until comparatively recently, acid requirements have been determined largely by a combination of empirical correlation and the experience of the operator in similar fields.

In order to remedy this situation and provide a theoretical basis from which estimates of permeability increase could be made, in terms of acid injected, a study was undertaken by Nougaro and Labbé in France. This study resulted in a comparatively simple equation, which furnished a linear relationship on a plot of the square root of the permeability against volumes of acid consumed. Experimental evidence in support of this theory was also given by these authors in their paper [1] from a laboratory study of a number of vesicular limestone samples.[1]

The choice of vesicular limestones is rather significant, for under low power magnification the macropore structure is easily visible and the pores appear to be of approximately uniform size. This uniformity of pore size is an important desideratum, for the theory is based on exactly this assumption.

Unfortunately, vesicular limestones such as those studied by Nougaro and Labbé do not constitute the major proportion of formations considered suitable for acid treatment. Consequently it was inevitable that subsequent laboratory investigations, upon other types of limestone, should produce anomalous results.

The discrepancies between laboratory tests and theoretical predictions are in some cases so serious

that it was considered desirable that a re-examination of the theory should be attempted.

The present study represents an attempt to reformulate a theory of acid treatment along somewhat more general lines than those proposed by Nougaro and Labbé. An assumption of uniformity in the effective pore structure seemed unnecessarily restrictive. Therefore, in the subsequent development of the theory a non-uniform pore model is taken as the basis. This, it is felt, is a more realistic representation of a limestone and also a more general one, for it includes the uniform pore system as a special case.

THEORETICAL DEVELOPMENT

In the following development of the theory of acid treatment, an idealized model of the porous medium is postulated. This model is assumed to consist of a bundle of capillaries, composed of a number of groups in each of which the individual capillaries have the same length and radius.[2, 3]

An identical model to the above is assumed as the basis of the mercury injection method for the determination of pore size distribution. Therefore, in conformity with this theory, the same symbolism will be adopted to describe the model. Thus the porous medium will be considered to be composed of N groups of pores, each containing n_i pores, radius r_i, and of length L_i. The fractional porosity of each group is ϕ_i, which is given by:

$$\phi_i = \frac{n_i \pi r_i^2}{A} \cdot \frac{L_i}{L}$$

where A and L are the area and length of the total sample respectively, and the suffix i ranges over the values (1, 2, 3, . . .) to distinguish the individual groups.

The rate of flow through any pore group i is given by Poiseuille's equation:

$$q_i = \frac{n_i \pi r_i^4}{8\mu L_i} \cdot \Delta P$$

$$= \frac{r_i^2 \phi_i A}{8\mu L_i^2} \cdot L \cdot \Delta P$$

and the total flow rate through all the pore groups is

$$Q = \sum_1^N q_i = \frac{A \cdot L \cdot \Delta P}{8\mu} \sum_1^N \frac{r_i^2 \phi_i}{L_i^2}$$

From Darcy's law:

$$Q = \frac{k_L \cdot A}{\mu \cdot L} \cdot \Delta P \cdot \frac{1}{1{\cdot}013 \times 10''}$$

where k_L is expressed in millidarcys.

Combining the above equations:

$$k_L = 0{\cdot}127 \times 10'' \sum_{i=1}^N \frac{r_i^2 \phi_i L^2}{L_i^2}$$

For a given length of sample this may be written as

$$k_L = C \sum_1^N \frac{r_i^2 \phi_i}{L_i^2} \quad \text{where } C = 0{\cdot}127 \times 10'' L^2$$

The acid treatment process, as applied to limestone formations, has for its object an increase in permeability. This is accomplished by dissolving the rock matrix to form an increase in effective pore space. In terms of the above model this may be interpreted as an increase in pore radius and a consequent increase in porosity. Quantitatively, the change in permeability may be determined from the above equation as follows.

If it is assumed, for simplicity, that L_i remains constant and equal to the mean pore length L_e and, furthermore, that each pore is acidized uniformly along its entire length, we may write for the permeability before acid treatment

$$k_{L1} = C \sum_1^N \frac{r_{i1}^2 \phi_{i1}}{L_e^2}$$

After acidizing

$$K_{L2} = C \sum_1^N \frac{r_{i2}^2 \phi_{i2}}{L_e^2}$$

also from

$$\phi_i = \frac{n_i \pi r_i^2}{A} \cdot \frac{L_e}{L}$$

$$\frac{\phi_{i1}}{\phi_{i2}} = \frac{r_{i1}^2}{r_{i2}^2}$$

from which

$$k_{L2} = C \sum_1^N \frac{r_{i1}^2 \phi_{i1}}{L_e^2} \left(\frac{\phi_{i2}}{\phi_{i1}}\right)^2$$

if $\Delta\phi_i = \phi_{i2} - \phi_{i1}$

$$k_{L2} = C \sum_1^N \frac{r_{i1}^2 \phi}{L_e^2} \left(1 + \frac{\Delta\phi}{\phi_{i1}}\right)^2$$

This formula gives the final permeability after acid treatment.

In laboratory investigations of acid treatment of limestone core plugs it has been customary, following the work of Nougaro and Labbé,[1] to present the results in the form of a plot of $k^{\frac{1}{2}}$ against volumes of acid used. To put the above equation in a form suitable for such a plot, it is necessary to relate the change in fractional porosity in any pore group to the volume of acid flowing through this group.

If x cc of limestone are dissolved per unit volume of spent acid and q_i volumes of acid flow through each pore group (assuming that all the acid reacts with the limestone), the change in porosity of group i will be:

$$\Delta\phi_i = \frac{q_i x}{V}$$

where V is the volume of the whole sample.

Hence $$k_{L2} = C \sum_1^N \frac{r_{i1}^2 \phi_{i1}}{L_e^2} \left(1 + \frac{q_i x}{V \phi_{i1}}\right)^2$$

and $$(k_{L2})^{\frac{1}{2}} = \left\{ C \sum_1^N \frac{r_{i1}^2 \phi_{i1}}{L_e^2} \left(1 + \frac{q_i x}{V \phi_{i1}}\right)^2 \right\}^{\frac{1}{2}}$$

Expanding the series:

$$(k_{L2})^{\frac{1}{2}} = \left\{ C \sum_1^N \frac{r_{i1}^2 \phi_{i1}}{L_e^2} + C \sum_1^N \frac{r_{i1}^2 \phi_{i1}}{L_e^2} \left(\frac{2q_i x}{V\phi_{i1}} + \left(\frac{q_i x}{V\phi_i} \right)^2 \right) \right\}^{\frac{1}{2}}$$

or

$$(k_{L2})^{\frac{1}{2}} = (k_{L1})^{\frac{1}{2}} \left\{ 1 + \frac{C}{k_{L1}} \sum_1^N \frac{r_{i1}^2 \phi_{i1}}{L_e^2} \left(\frac{2q_i x}{V\phi_{i1}} + \left(\frac{q_i x}{V\phi_{i1}} \right)^2 \right) \right\}^{\frac{1}{2}}$$

If in the above equation $\frac{q_i x}{V\phi_{i1}}$ has the same value for all i the above formula reduces to

$$(k_{L2})^{\frac{1}{2}} = (k_{L1})^{\frac{1}{2}} \left(1 + \frac{q_i x}{V\phi_{i1}} \right)$$

or

$$(k_{L2})^{\frac{1}{2}} = (k_{L1})^{\frac{1}{2}} \left(1 + \frac{\Delta\phi_i}{\phi_{i1}} \right)$$

To assess the implication of the assumption that $\frac{q_i x}{V\phi_{i1}}$ or $\frac{\Delta\phi_i}{\phi_{i1}}$ is constant, we find that:

$$\frac{\Delta\phi i}{\phi_{i1}} = \text{const or } \Delta\phi_i = M\phi_{i1}, \text{ say,}$$

or the change in porosity is proportional to the fractional porosity of the group.

If $\Delta\phi$ is the total change in porosity of the sample and ϕ the total fractional porosity, then

$$\Delta\phi = \sum \Delta\phi_i \text{ and } \phi_1 = \sum \phi_{i1}$$

from which it follows that

$$\Delta\phi = M\phi_1$$

and

$$\frac{\Delta\phi_i}{\phi_{i1}} = \frac{\Delta\phi}{\phi_1}$$

Similarly, it may be demonstrated that

$$\frac{q_i}{\phi_{i1}} = \frac{Q}{\phi_1}$$

where Q is the total volume of acid reacted.

This means that the volume flow is proportional to the porosity. But from the basic premise of the validity of the Poiseuille equation

$$q_i = \frac{r_i^2 \phi_i A \,.\, L \,.\, \Delta P}{8\mu L_e^2}$$

if we consider two pore groups i and j, and the pressure drop across each pore group is the same,

$$\frac{q_i}{q_j} = \frac{r_i^2 \phi_i}{r_j^2 \phi_j} \quad \text{and} \quad \frac{q_i}{Q} = \frac{r_i^2 \phi_i}{\bar{r}^2 \phi}$$

It is obvious that the flow through each pore group can only be proportional to the porosity if:

$$r_i^2 = r_j^2 = \bar{r}^2$$

i.e. if all the pores are of equal radius.

The expression for the above particular case may therefore be written as:

$$(k_{L2})^{\frac{1}{2}} = (k_{L1})^{\frac{1}{2}} \left(1 + \frac{Qx}{V\phi_1} \right)$$

or

$$(k_{L2})^{\frac{1}{2}} = (k_{L1})^{\frac{1}{2}} \left(1 + \frac{\Delta\phi}{\phi_1} \right)$$

This is found to be the Nougaro and Labbé equation (as derived in Appendix II), which appears as a special case of the more general theory, valid only for samples in which the pores are of uniform size.

For samples in which there is a distribution of pore size the more general equation must be used, *i.e.* $(k_{L2})^{\frac{1}{2}}$ must be calculated from:

$$(k_{L2})^{\frac{1}{2}} = \left\{ C \sum_1^N \frac{r_{i1}^2 \phi_{i1}}{L_e^2} \left(1 + \frac{q_i x}{V\phi_{i1}} \right)^2 \right\}^{\frac{1}{2}}$$

APPLICATION OF THE THEORY

In order to apply the equations developed in the previous section to the calculation of the curve representing the increase of permeability due to acidizing it is essential that certain fundamental information be available. This is:

1. The pore size distribution curve as determined by the mercury injection method.
2. The total volume of the sample.
3. The value of the constant x, which represents the volume of limestone dissolved per unit volume of acid used.

The calculations are then organized in an incremental or stepwise manner according to the following scheme.

Considering first the pore size distribution curve. This is a continuous curve representing a continuous distribution of pore radii, which must be approximated by a number of groups of pores of constant radius. The problem is identical to that of approximate integration, in which the area under a continuous curve is approximated by a series of rectangles, as in a mid-ordinate method, the condition being that the areas under the continuous and approximate curves be the same. From this finite difference approximation the group radii, r_i, and fractional porosities, ϕ_i, are calculated.

The total amount of acid to be injected is subdivided into increments of equal volume and the injection is assumed to be conducted in stages, the permeability after each stage being calculated from:

$$k_{L2} = C \sum_1^N \frac{r_{i1}^2 \phi_{i1}}{L_e^2} \left(1 + \frac{q_i x}{V\phi_{i1}} \right)^2$$

in which q_i is determined as follows.

From the equation for the flow through any pore group i,

$$q_i = \frac{r_i^2 \phi_i A \,.\, L \,.\, \Delta P}{8\mu L_e^2}$$

and

$$Q = \sum_1^N q_i = \frac{AL\Delta P}{8\mu L_e^2} \sum_1^N r_i^2 \phi_i$$

$$\frac{q_i}{Q} = \frac{r_i^2 \phi_i}{\sum r_i^2 \phi_i}$$

If the porosities are expressed as a percentage of the total pore space, then

$$\frac{\phi_i}{\phi_t} \times 100 = S_i$$

and the above equation becomes:

$$q_i = Q \cdot \frac{r_i^2 S_i}{\sum r_i^2 S_i}$$

The values of ϕ_{i2} and r_{i2} may be calculated from the formulæ

$$\phi_{i2} = \phi_{i1}\left(1 + \frac{\Delta\phi_i}{\phi_{i1}}\right)$$

where $$\Delta\phi_i = \frac{q_i x}{V}$$

and $$r_{i2}^2 = r_{i1}^2\left(1 + \frac{\Delta\phi_i}{\phi_{i1}}\right)$$

Utilizing these values in the equation

$$k_{L2} = C\sum_1^N \frac{r_{i1}^2\phi_{i1}}{L_e^2}\left(1 + \frac{q_i x}{V\phi_{i1}}\right)^2$$

in a stepwise manner, the permeability curve may be defined. A numerical example demonstrating the method is given, the results appearing in Table I and Figs 1–5.

DISCUSSION OF RESULTS

The present discussion of the results of this study will be based upon the calculations made for the typical limestone core treated in the numerical example. It may perhaps be argued that such a sample is hardly representative of the type of formation best suited to acid treatment. This may be true, but the choice was not governed entirely by such considerations. The theory proposed in this report is expressly designed for limestones in which there is a distribution of pore size, a condition fulfilled by limestone core No. 11.* It is felt that any conclusions drawn from considerations based on this sample will retain their validity for other samples, no matter what the form of the distribution, differences being in degree rather than in principle.

The curve of Fig 1 shows the pore size distribution as obtained by the mercury injection method, together with the values given by a relatively coarse approximation of seven pore groups. Permeabilities calculated from the original curve and the approximation are 11·0 mD and 10·44 mD respectively, which, in view of the reasonable agreement between the curves, may be considered satisfactory for most practical purposes.

For the value of x, the volume of limestone dissolved per unit volume of acid reacted, a theoretical value was calculated from the equation:

$$CaCO_3 + 2HCl = CaCl_2 + H_2O + CO_2$$

the unit volume of N/100 HCl being taken as 1 litre.

On this basis the final permeability after each stage of acidizing with successive unit volumes of N/100 was

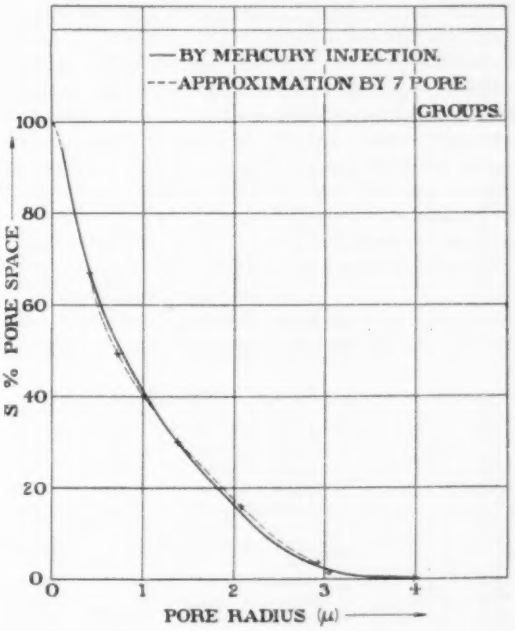

FIG 1
PORE SIZE DISTRIBUTION

calculated as in Table I. The results of this calculation for both the present theory and the Nougaro and Labbé theory are plotted in Fig 2, in the form $(k)^{\frac{1}{2}}$ against volumes of acid. In order to apply the latter theory, the uniform pore radius was taken as the mean pore radius of the distribution. This procedure is perhaps open to the objection that undue weight has been placed on the contribution of the smaller pores in the calculation of the mean radius. These pores, under the influence of any given pressure gradient, would play only a relatively minor role in the flow process; therefore it might be permissible to assume an arbitrary "cut-off" radius and perform the calculation only on the larger pores. In this case the mean radius would undoubtedly be greater, but the slope of the curve would not be appreciably different. Furthermore, similar considerations must also apply equally to the calculation based on the distribution of sizes, the net trends remaining unaltered.

This point is illustrated by the curves plotted in

* The properties of this core are given in Appendix I.

TABLE I

Calculations for Limestone Core No. 11

Group	r_i, μ	ϕ_{i1}, $\times 10^{-2}$	S_i, %	r_{i1}^2	$r_{i1}^2 S_{i1}$	q_i, %	$\Delta\phi_i$ $\times 10^{-2}$	ϕ_{i2} $\times 10^{-2}$	$\frac{\Delta\phi_i}{\phi_{i1}}$	r_{i2}^2	$\bar{k}$, mD
1	3·51	0·268	1·0	12·2	12·30	7·0	0·142	0·410	0·530	18·80	
2	3·0	0·805	3·0	9·0	27·00	15·3	0·318	1·230	0·395	12·55	
3	2·52	2·01	7·5	6·35	47·60	27·0	0·562	2·570	0·280	8·13	
4	1·80	5·09	19·0	3·24	61·50	34·9	0·726	5·820	0·143	3·70	*10·44*
5	1·05	5·09	19·0	1·103	20·25	11·9	0·248	5·34	0·0487	1·156	
6	0·575	4·69	17·5	0·331	5·80	3·3	0·069	4·76	0·0147	0·336	
7	0·20	8·85	33·0	0·04	1·32	0·75	0·016	8·86	0·0018	0·0401	
		=26·79	100·0		176·50	100·15	2·071	28·99			
1		0·410	1·415	18·80	26·8	10·51	0·219	0·629	0·533	28·85	
2		1·23	4·25	12·55	53·4	20·93	0·435	1·665	0·362	17·10	
3		2·57	8·87	8·13	72·1	28·30	0·589	3·159	0·229	9·99	
4		5·82	20·10	3·70	74·5	29·22	0·609	6·429	0·1096	4·08	*16·23*
5		5·34	18·45	1·156	21·3	8·35	0·174	5·514	0·0326	1·19	
6		4·76	16·45	0·[illegible]3	5·56	2·18	0·045	4·805	0·0095	0·339	
7		8·86	30·60	0·0401	1·23	0·48	0·010	8·870	0·0011	0·0403	
		=28·99	100·135		254·89	99·97	2·091	31·071			
1		0·629	2·025	28·85	58·45	16·04	0·334	0·963	0·531	44·20	
2		1·665	5·370	17·10	91·80	25·20	0·525	2·190	0·315	22·50	
3		3·159	10·18	9·99	101·70	27·92	0·580	3·739	0·184	11·83	
4		6·429	20·71	4·08	84·50	23·20	0·483	6·912	0·075	4·39	*24·9*
5		5·514	17·78	1·19	21·15	5·81	0·121	5·635	0·0219	1·218	
6		4·805	15·50	0·339	5·25	1·44	0·030	4·835	0·0062	0·341	
7		8·870	28·60	0·0403	1·15	0·316	0·007	8·877	0·0008	0·0403	
		31·071	100·165		364·00	99·926	2·07	33·149			
1		0·963	2·91	44·2	128·50	24·35	0·506	1·469	0·525	67·40	
2		2·190	6·61	22·5	148·70	28·20	0·586	2·776	0·268	28·52	
3		3·739	11·30	11·83	133·80	25·30	0·526	4·265	0·1405	13·50	
4		6·912	20·85	4·39	91·50	17·33	0·360	7·272	0·052	4·62	*38·45*
5		5·635	17·00	1·218	20·70	3·92	0·082	5·717	0·0146	1·235	
6		4·835	14·60	0·341	4·98	0·755	0·016	4·851	0·0033	0·342	
7		8·877	26·70	0·0403	1·08	0·0001	—	8·877	—	0·0403	
		33·149	99·97		529·26	99·86	2·076	35·227			
1		1·469	4·17	67·40	281·0	35·50	0·739	2·208	0·503	101·30	
2		2·776	7·90	28·52	225·0	28·41	0·591	3·367	0·213	34·60	
3		4·265	12·13	13·50	164·0	20·73	0·431	4·696	0·1004	14·86	*61·3*
4		7·272	20·65	4·62	95·5	12·08	0·251	7·523	0·0346	4·88	
5		5·717	16·25	1·235	20·05	2·53	0·053	5·770	0·0093	1·247	
6		4·851	13·80	0·342	4·72	0·597	0·012	4·863	0·0025	0·343	
7		8·877	25·10	0·0403	1·01	—	—	8·877	—	—	
		35·227	100·00		791·28	99·847	2·077	37·304			
1		2·208	5·93	101·30	601·0	49·2	1·023	3·231	0·464	148·5	
2		3·367	9·04	34·60	312·6	25·5	0·531	3·898	0·158	40·1	
3		4·696	12·60	14·86	187·2	15·3	0·318	5·014	0·0677	15·84	
4		7·523	20·20	4·88	98·7	8·06	0·168	7·691	0·0223	5·0	*100·4*
5		5·770	15·50	1·247	19·3	1·58	0·033	5·803	0·0057	1·253	
6		4·863	13·05	0·343	4·5	0·37	0·008	4·871	0·0016	0·343	
7		8·877	23·80	0·0403	1·0	—	—	8·877	—	—	
		37·304	99·92		1224·3	100·01	2·088	39·385			

Fig 3. Here the curves numbered 1–7 represent the results that would be obtained if each of the pore groups 1–7 occupied the whole of the pore space. In effect, these curves describe seven different uniform porous media, the initial pore radii of which are

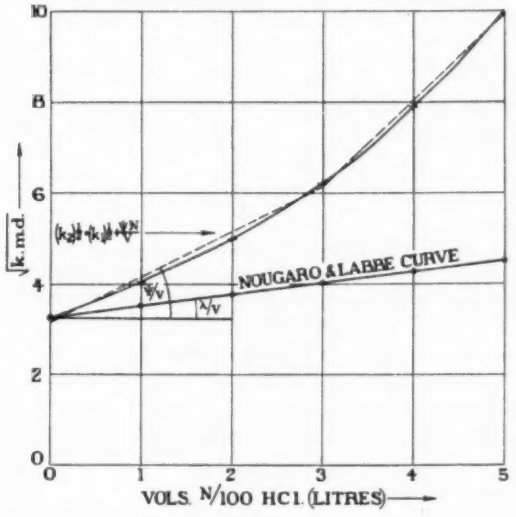

FIG 2

INCREASE IN PERMEABILITY IN NON-UNIFORM SAMPLE DUE TO ACIDIZING

identical with the initial radii of the seven pore groups.

It is obvious from theoretical considerations that these curves obey the Nougaro and Labbé equation. The slopes of the curves increase with increasing initial pore radius as would be expected, the ratio of the slopes of any two curves being proportional to the square root of the ratio of the respective permeabilities.

Equally obvious is the fact that the calculated curve for a non-uniform sample does not obey this equation. The curve for this sample, also drawn on Fig 3, differs in several important aspects:

1. It is non-linear and of continuously increasing slope.
2. The slope at every point is greater than that predicted by the Nougaro and Labbé equation.
3. The slope at every point is greater than that of any of the constituent pore groups, viewed as uniform media.

These observations are perhaps hardly surprising and qualitatively one might ascribe the deviation from linearity as being due to "breakthrough." In considerations of actual limestones, this would imply a gradual increase in size of the larger pores along the flow path, culminating in a relatively sudden emergence of the enlarged section at the outflow face. In this way rapid increases in permeability are possible.

In actual limestones such an explanation of observed trends of this nature might well be true. However, the present calculations are based on an idealized model in which such conditions are precluded, by virtue of the restricting assumption that each pore is acidized uniformly along its entire length. Hence a different explanation is required which is not incompatible with both model and actual formation.

A preliminary examination of the results in Table I will indicate that the groups of larger pores take an increasing percentage of the total flow as acidizing proceeds. The increase, however, is not maintained uniformly, for eventually only the group containing the largest pores takes an increasing percentage, and this is at the expense of all smaller pores. Thus the qualitative description of the curve is that the larger pores become dominant in the flow pattern, a state somewhat akin to "breakthrough." To present a quantitative description, more satisfactory from a theoretical point of view, the model must be viewed in a slightly different manner.

The model, as postulated, consisted of a number of pore groups each containing pores of uniform radius.

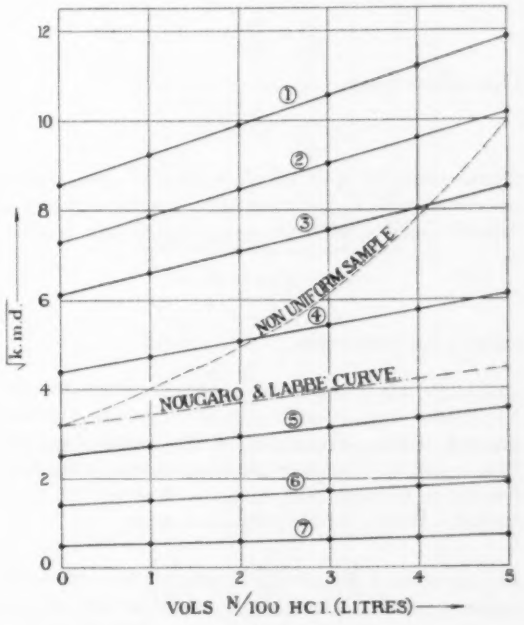

FIG 3

PERMEABILITY INCREASE IN EQUIVALENT UNIFORM MEDIA

Each group considered as an entity has a fractional porosity and a permeability, and the total of all pores of a given size may be associated with a definite area of the cross-section of the whole sample. In this way the sample appears as an assemblage of sections of different permeability arranged in parallel, or a special case of the problem of permeability stratification.

The mean permeability of the total sample may then be calculated from the knowledge of the constituent permeabilities and the respective flow areas.

To calculate these values for a non-uniform pore sample it will be necessary to consider at the same time the corresponding values for uniform pore samples. Thus it is proposed to employ subscripts u and nu when referring to the respective values for uniform and non-uniform samples. For example, k_{iu} will refer to the permeability of a uniform porous medium, the initial pore radius of which is r_i, and k_{inu} will refer to the permeability of pore group i in the non-uniform sample, the initial radius of which is r_i.

Consider two samples of a limestone porous medium of identical dimensions, one composed of uniform pores, the other of groups of pores of different pore size. Attention will be focused on the pore group i having, initially, pores of the same radius as the uniform medium. If the uniform medium has a total of N_i pores and a porosity Φ_i, the pore density or tube density being constant and equal to d_i, we have for the fractional porosity ϕ_i corresponding to n_i pores:

$$\frac{n_i}{N_i} = \frac{\phi_i}{\Phi_i}$$

This follows from

$$n_i = \frac{\phi_i A}{\pi r_i^2} \cdot \frac{L}{L_e}$$

Furthermore, if a_i is the flow area of cross-section associated with n_i pores, the tube density d_i, which is defined as the number of pores per unit area, is given by

$$d_i = \frac{n_i}{a_i} = \frac{N_i}{A}$$

Darcy's law states that

$$q_i = a_i \frac{k_i}{\mu} \cdot \frac{\partial P}{\partial L}$$

where k_i is the permeability of the uniform sample. This equation is valid for the area a_i because the tube density is uniform and equal to that of the total sample. Hence, for the uniform sample

$$\left(\frac{q_i}{Q}\right)_u = \left(\frac{a_i}{A}\right)_u = \left(\frac{\phi_i}{\Phi_i}\right)_u$$

Consider now the pore group i of the non-uniform sample. This group is composed of uniform pores of radius r_i. It is further postulated that the initial permeability of this group is identical to that of the equivalent uniform sample.

In conformity with the requirements laid down for the applicability of Darcy's law to a fractional area, in the case of a uniform sample, it is obvious that the necessary condition for equal permeability is that the tube densities shall be equal.

If n_i' and a_i' are the number of pores and associated flow area of cross-section, respectively, for pore group i.

$$\frac{n_i'}{a_i'} = \frac{n_i}{a_i} = \frac{N_i}{A} = d_i$$

also, from

$$n_i = \frac{\phi_i}{\pi r_i^2} \cdot \frac{AL}{L_e}$$

$$\frac{\phi_i}{n_i} = \frac{\phi_i'}{n_i'}$$

where ϕ_i' is the fractional porosity of group i. This may be written as

$$\frac{\phi_i}{a_i d_i} = \frac{\phi_i'}{a_i' d_i}$$

or

$$\frac{\phi_i}{a_i} = \frac{\phi_i'}{a_i'}$$

Now, letting $\phi_i' = \phi_i$; $a_i' = a_i$ and taking the initial condition, for which ϕ_i and Φ_i are represented by ϕ_{i1} and Φ_{i1} respectively, it follows that

$$\left(\frac{a_i}{A}\right)_{nu} = \left(\frac{a_i}{A}\right)_u = \frac{\phi_{i1}}{\Phi_{i1}} = S_{i1}$$

This expression determines the flow area of cross-section associated with any pore group i, in terms of an equivalent uniform medium. This area will remain constant.

From the theory of permeability stratification the mean permeability of a porous medium, stratified in a direction parallel to the direction of flow, is given by

$$\bar{k} = \frac{\sum a_i k_i}{\sum a_i} = \sum \frac{a_i}{A} k_i$$

which becomes in the present case

$$\bar{k} = \sum_1^N S_{i1} k_i$$

After m volumes of acid have flowed through the sample

$$\bar{k}_m = \sum_1^N S_{i1} k_{im}$$

where k_{im} is the permeability of pore group i after m volumes have flowed through the sample.

From the equation

$$q_i = \frac{r_i^2 \phi_i A}{8 \mu L_e^2} \cdot L \cdot \Delta P$$

$$\frac{q_{im}}{Q} = \frac{r_{im}^2 \phi_{im}}{\sum r_{im}^2 \phi_{im}} = \frac{r_{im}^2 \phi_{im}}{\bar{r}_m^2 \phi_m}$$

also

$$q_{im} = a_i \frac{k_{im}}{\mu} \cdot \frac{\partial P}{\partial L}$$

$$Q = A \frac{\bar{k}_m}{\mu} \cdot \frac{\partial P}{\partial L}$$

or

$$\frac{q_{im}}{Q} = \frac{a_i}{A} \cdot \frac{k_{im}}{\bar{k}_m} = S_{i1} \frac{k_{im}}{\bar{k}_m}$$

and as

$$\bar{k}_m = C' \bar{r}_m^2 \phi_m$$

$$k_{im} = \frac{C' r_{im}^2 \phi_{im}}{S_{i1}}$$

But, for the same pressure gradient

$$\frac{q_{im}}{q_{i1}} = \frac{r_{im}^2\phi_{im}}{r_{i1}^2\phi_{i1}} = \frac{k_{im}}{k_{i1}}$$

and it has been shown previously that

$$\left(\frac{r_2}{r_1}\right)^2 = \left(\frac{\phi_2}{\phi_1}\right)$$

Hence $$\frac{k_{im}}{k_{i1}} = \left(\frac{r_{im}}{r_{i1}}\right)^4$$

from which $$\bar{k}_m = \sum_1^N S_{i1}k_{i1}\left(\frac{r_{im}}{r_{i1}}\right)^4$$

or $$\bar{k}_m = \sum_1^N S_{i1}k_{i1}\left(1 + \frac{\Delta\phi_{im}}{\phi_{i1}}\right)^2$$

Thus we have an alternative form of the equation for calculating the permeability of the sample at any stage of the acidizing process, expressed in terms of the permeabilities of the various groups.

This formulation has the advantage that it is now possible to calculate the variation in permeability in each pore group, in terms of acid injected. The equivalence of the two forms of the equation is easily demonstrated, for

$$k_{i1} = C'r_{i1}^2\Phi_{i1}$$

and $$S_{i1} = \frac{\phi_{i1}}{\Phi_{i1}}$$

hence $$S_{i1}k = C'r_{i1}^2\phi_{i1} = \frac{C}{L_e^2}r_{i1}^2\phi_{i1}$$

from which

$$\sum_1^N S_{i1}k_{i1}\left(1 + \frac{\Delta\phi_{im}}{\phi_{i1}}\right)^2 = C\sum_1^N \frac{r_{i1}^2\phi_{i1}}{L_e^2}\left(1 + \frac{\Delta\phi_i}{\phi_{i1}}\right)^2$$

which is the form in which the equation was originally derived.

The difficulty in the application of the alternative form of the equation lies in the specification of the initial group permeability which, in general, in the case of a non-uniform limestone sample, is unknown. This difficulty does not arise if these concepts are applied to the field case of a stratified limestone formation for which the constituent porosities and permeabilities may be determined.

Writing the equation in a slightly different manner as

$$\bar{k}_m = \sum_1^N \phi_{i1}\frac{k_{i1}}{\Phi_{i1}}\left(1 + \frac{\Delta\phi_{im}}{\phi_{i1}}\right)^2$$

where $$k_{i1} = C'r_{i1}^2\Phi_{i1}$$

and $$\left(\frac{k_{i1}}{\Phi_{i1}}\right) \propto r_{i1}^2$$

If either k_{i1} or Φ_{i1} were known, the other would be determined uniquely for a given value of r_i. As neither is known, the choice is to some extent arbitrary, for, whatever values are selected, the ratio k_{i1}/Φ_{i1} must remain constant at the value determined by r_{i1}.

In a non-uniform sample r_{i1} and ϕ_{i1} may be considered known; hence, for the purposes of illustrating the variations in group permeability an arbitrary Φ_{i1} may be assumed, from which the corresponding k_{i1} is calculated. The actual value of Φ_{i1} is of no great importance, for it merely determines the initial value of the group permeability and does not affect either the subsequent trend of the curves or the conclusions to be drawn from them.

For this reason a convenient value of Φ_{i1} was adopted in this study, *i.e.* Φ_{i1} was assumed to be the same for each pore group and equal to the initial total porosity, ϕ_1, of the non-uniform sample. With this assumed value the respective k_{i1} values for the individual pore groups are given by

$$\frac{k_{i1}}{\bar{k}_1} = \frac{r_{i1}^2}{\bar{r}_1^2}$$

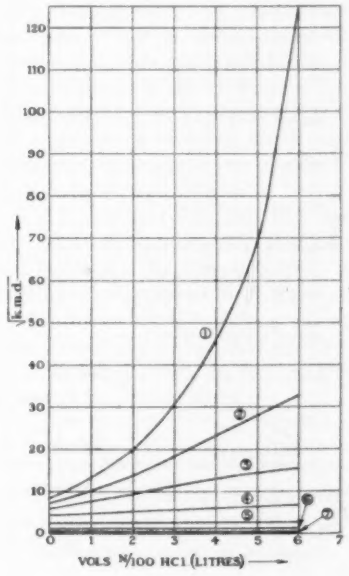

FIG 4

INCREASE IN GROUP PERMEABILITY

From this initial permeability distribution in the pore groups, the variation of group permeability in terms of acid injected may be calculated. The curves showing this variation are given in Figs 4 and 5.

Having determined the actual permeability variation in the separate groups, we are now in a position to interpret the results in a more satisfactory manner.

Consider first the curves of Figs 3–5, in which several pertinent features are readily apparent. For example, Figs 3 and 4 are plotted in the same coordinate system for the purposes of comparison of the group permeabilities with those of uniform media. It will be observed that the permeabilities of the groups

of larger pores increase at a much greater rate than those of the corresponding uniform samples. On the other hand, for the smaller pores the uniform media increase more rapidly than the small pore groups. The explanation of this tendency may be sought in the numerical values of Table I, in which it will be found that the larger pore groups take a much higher percentage of the total acid injected than would be the case for a uniform sample and conversely for the smaller pores. A qualitative picture would suggest by-passing through the larger pores due to constrictions imposed by the smaller ones.

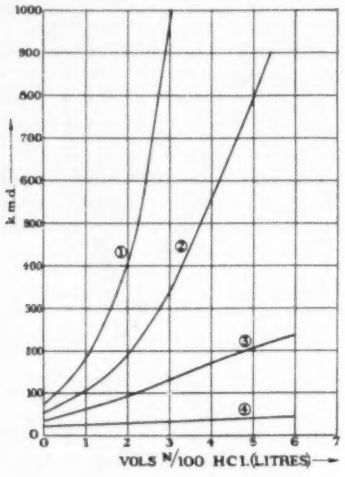

FIG 5

INCREASE IN GROUP PERMEABILITY

A more explicit formulation may be given in terms of the concept of uniform and non-uniform samples previously treated. It was found that

$$\left(\frac{q_i}{Q}\right)_{nu} = \frac{r_i^2\phi_i}{\bar{r}^2\phi}$$

For a uniform sample $r_i = \bar{r}$ and

$$\left(\frac{q_i}{Q}\right)_{u} = \frac{\phi_i}{\Phi_i}$$

But in the calculations Φ_i has been assumed equal to ϕ.

Hence $$(q_i)_{nu} = (q_i)_u \cdot \frac{r_i^2}{\bar{r}^2}$$

and $$(q_i)_{nu} \gtrless (q_i)_u \text{ as } r_i \gtrless \bar{r}$$

Therefore for all groups in which $r_i > \bar{r}$, we should expect the group permeability to increase more rapidly than in the corresponding uniform medium and conversely for groups with $r_i < \bar{r}$. As $\bar{r}$ increases due to acidizing, the initial trends cannot be maintained, for the mean value is influenced to a much greater extent by the very rapid increase in the radii of the groups of largest pores. It is inevitable therefore that more and more of the groups enter the class for which $r_i < \bar{r}$, and the evidence of this is the decrease in slope of their permeability curves. It is of interest to note that, at the stage when $r_i = \bar{r}$, the slopes of the curves for the group permeability and uniform permeability are identical.

In one case only does the permeability increase uniformly; this is in the group of the largest pores, for the radius of this group must always be greater than the mean radius. Thus ultimately it is the largest pores alone which dominate the process, contributing the greatest proportion of the total increase in permeability.

Another significant observation might be made from a study of the curves of Figs 4 and 5. This is the approximate linearity of the initial portions of the group permeability curves. In the example treated, all the curves are approximately linear up to the stage at which 1·5 vol of acid have been injected. Hence, as the total permeability is a linear combination of the group permeabilities, we should expect the curve for the total sample (Fig 2) to be linear up to this point, which is indeed the case.

One final observation must be made. This relates to the contribution to total permeability of the groups of the largest pores. Initially, in the numerical example, the four groups of larger pores had radii $r_i > \bar{r}$, and calculations show that these pores contribute 84 per cent of the total permeability. After three volumes of acid had been injected the contribution of these groups had risen to 95 per cent of the total. At this stage the increment from group 4 was already declining, but the numerical values suffice to illustrate that the total permeability curve must have a slope greater than could be described by any assumption of uniformity of pore structure. In other words, in a sample in which there is a distribution of pore size the increase in permeability due to acidizing cannot be described by the existing equation of Nougaro and Labbé. Even in the initial linear portion of the curve the slope must be greater than that predicted by this equation, for the reasons stated above.

Comparison of the theory proposed in this study with available laboratory investigations is considerably hampered by the fact that the relevant pore size distribution data were not determined. Thus no more than qualitative agreement could be expected, and even this must be of a rather tentative nature.

Perhaps the most significant observation to be made upon the results of these investigations is the frequent occurrence of non-linearity and discontinuity in the permeability curves. The discontinuities result from drawing straight lines through the experimental curves rather than curves, in an attempt to verify the Nougaro and Labbé equation. It is quite possible to produce a similar discontinuity in the results of the present study by approximating the curve with two straight

lines. The result, however, is meaningless and misleading, for the curvature is capable of a quite different explanation. Claims that an initial linear portion verifies this equation are erroneous, for in the light of the present study any measure of deviation from linearity is indicative of non-uniformity in the effective pore structure. Even an approximately linear plot must be viewed with circumspection if it is intended to extrapolate beyond the range of the laboratory tests, for it may merely mean that acidizing had not been carried far enough. This may be seen from Fig 2 for an approximately linear relationship up to 3 vol of acid. In this case the increase in permeability is from 10·4 to 37·7 mD. Extrapolation to 5 vol of acid would indicate a permeability of 61·5 mD instead of the 100 mD actually calculated.

Such laboratory investigations are of value, however, irrespective of any theoretical interpretation, for, providing the required permeability range is covered in the tests, a reasonably reliable guide to acid requirements should be obtained.

CONCLUSIONS

The conclusions derived from this study may be summarized as follows.

It has been found that the existing theory of acid treatment, based on uniformity of pore structure, is of limited validity and as such should be applied with caution. In this respect, it is desirable that supplementary information regarding the pore size distribution should be obtained wherever possible to aid the interpretation of laboratory tests. This information would be given either by microscopic examination or by routine pore size distribution measurements, depending on the formation under investigation.

Laboratory investigations conducted on core plugs provide valuable estimates of the permeability increases to be expected, providing that the whole range of interest is covered. It is preferable to extend the investigation beyond the range of immediate interest in cases in which non-uniformity of effective pore structure is suspected. Indications of such non-uniformity should manifest themselves in non-linearity of the experimental curves when plotted on the basis of $(k)^{\frac{1}{2}}$ against volumes of acid spent.

The reason for extending the range of the investigation is to place limits on any linear approximation adopted, to provide semi-empirical coefficients for use in Nougaro and Labbé type equations

$$(k_2)^{\frac{1}{2}} = (k_1)^{\frac{1}{2}} + \frac{\psi N}{V}$$

The term semi-empirical is used with reason, for any linear approximation to a non-uniform sample is without theoretical foundation and cannot be used to estimate properties of the medium, such as mean effective pore radius.

It is suggested that further work should be done to determine that portion of the pore size distribution which may be classed as "effective." It has been shown that the larger pores alone contribute the major portion of the permeability, and for practical applications it would be desirable to determine whether some form of cut-off radius could be assumed, below which the smaller pores play no useful role. Such knowledge would have important bearing on calculations of acid requirements for given increases in permeability.

The presence of a distribution of effective pore size appears to be beneficial to the process of acid treatment in reasonably homogeneous (*i.e.* non-fissured) limestone formations. This is reflected in the much greater increase in permeability created in the larger pore groups than would be expected from calculations based on mean overall pore properties. With regard to vesicular and fissured systems, these may be considered as limiting cases of pore size distributions with perfectly well defined cut-off radii.

In view of the great contribution of the larger pore groups to the total permeability and the rapid increases in permeability attained in these groups by acidizing, it is suggested that attempts to block them off, for the purposes of more uniform acidizing of the whole medium, must result in a less economical employment of the acid. It is felt that on the contrary these tendencies should be encouraged.

ACKNOWLEDGMENT

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Note: A comprehensive list of references relating to capillary models of porous media is given by: Scheidegger, A. E. "The Physics of Flow through Porous Media." Toronto: University of Toronto Press, 1957.

APPENDIX I

Properties of Limestone Core No. 11 and Values of the Constants Used in the Calculation

Bulk volume of plug $V = 9{\cdot}61$ cc
Volume of pore space = 2·58 cc
Porosity = 26·8 per cent rock volume
Permeability calculated from P.S.D. = 11·0 mD
Assumed limestone density = 2·62 g/cc
Value of x vol of limestone dissolved/unit vol of N/100 HCl
0·2 cc $CaCO_3$/litre N/100 HCl
$x/V = 0{\cdot}0208$/litre N/100 HCl
Calculations based on an injection of 1 litre of N/100 HCl

APPENDIX II

The Nougaro and Labbé Equation

This appendix presents a simplified derivation of an equation originally proposed by Nougaro and Labbé [1] to describe the

increase in permeability in a limestone formation due to acidizing. The purpose of this derivation is to demonstrate the complete equivalence of the equation appearing as a special case in this report with that of Nougaro and Labbé, which is given in a somewhat different form.

Consider a limestone sample composed of pores of uniform size. Calculations based on pore size distribution indicate that the permeability may be expressed as

$$k = C\bar{r}^2\phi \qquad \text{where } C = \frac{1{\cdot}27}{(L_e/L)^2}$$

As the pores are uniform $r = \bar{r}$, therefore initially

$$k_1 = Cr_1^2\,\phi_1$$

After acid treatment the permeability is k_2 and

$$k_2 = Cr_2^2\phi_2$$

from which

$$\frac{k_2}{k_1} = \frac{r_2^2\phi_2}{r_1^2\phi_1}$$

It has been shown that

$$\left(\frac{r_2}{r_1}\right)^2 = \frac{\phi_2}{\phi_1}$$

therefore

$$\frac{k_2}{k_1} = \left(\frac{\phi_2}{\phi_1}\right)^2 = \left(1 + \frac{\Delta\phi}{\phi_1}\right)^2$$

and

$$(k_2)^{\frac{1}{2}} = (k_1)^{\frac{1}{2}}\left(1 + \frac{\Delta\phi}{\phi_1}\right) = (k_1)^{\frac{1}{2}}\left(1 + \frac{Qx}{V\phi_1}\right)$$

which is the form of equation given in this report.

The equation presented by Nougaro and Labbé is

$$(k_2)^{\frac{1}{2}} = (k_1)^{\frac{1}{2}} + \frac{\lambda N}{V}$$

where N is a number proportional to the mol of acid reacted;
V is the bulk volume of the sample;
λ is a constant representing the susceptibility of the rock to acidizing.

The value of λ is given as:

$$\lambda = \left(\frac{v^2}{8\pi\alpha^3\beta}\right)^{\frac{1}{2}}$$

where v is the volume of rock dissolved per N units of acid;
α is the tortuosity (L_e/L);
β is the number of channels per unit cross-section of flow area.

For the two flow equations to be equivalent we must have

$$\lambda = \frac{(k_1)^{\frac{1}{2}}x}{\phi_1}$$

or

$$\lambda^2 = \frac{k_1x^2}{\phi_1^2} = \frac{x^2/\phi_1^2}{k_1}$$

Now

$$\phi_1 = \frac{N\pi r_1^2L_e}{AL}$$

and

$$Q = \frac{AL\Delta Pr_1^2\phi_1}{8\mu L_e^2}$$

From Darcy's law:

$$Q = \frac{k_LA\Delta P}{\mu L}\cdot\frac{1}{1{\cdot}013\times10''}$$

or

$$k_L = \frac{r_1^2\phi_1L^2}{8L_e^2}\cdot 1{\cdot}013\times10''\text{ md}$$

Put $1{\cdot}013\times10'' = c'$

and

$$\frac{\phi_1^2}{k_1} = \frac{N^2\pi^2r_1^4L_e^2}{A^2L^2}\cdot\frac{8L_e^2}{r_1^2\phi_1L^2c'} = \frac{N^2\pi^2r_1^4L_e^48}{A^2L^4r_1^2c'}\cdot\frac{A\,.\,L}{N\pi r_1^2L_e}$$

or

$$\frac{\phi_1^2}{k_1} = \frac{8N\pi L_e^3}{c'AL^3}$$

Therefore

$$\frac{x^2/\phi_1^2}{k_1} = \frac{x^2}{\dfrac{8N\pi}{c'A}\cdot\left(\dfrac{L_e}{L}\right)^3}$$

Transposing this equation into the symbols adopted by Nougaro and Labbé

$$x = v : \frac{N}{A} = \beta : \frac{L_e}{L} = \alpha$$

then

$$\frac{x^2}{\phi_1^2/k_1} = \frac{c'v^2}{8\pi\alpha^3\beta} = c'\lambda^2$$

In the Nougaro and Labbé equation, the permeability is given in Darcys. The constant c' is thus implied in the units employed for measuring the pressure and viscosity, and hence does not appear. If in the present study this convention is adopted, the two equations become identical.

DISCUSSION

Robert J. Holley: The author has mentioned in his paper that the "acidizing" process has been successfully applied in the field for many years with tangible results. In fact, two chemists, Herman Frasch and John W. Van Dyke, are on record as having improved the productivity of wells in the famous Lima field of Ohio by acidizing the dolomitic Trenton Limestone formation in 1895. By 1896 claims were made that their method was beyond the experimental stage and that production could be increased where all other methods failed (*Derrick* magazine of Oil City, Pennsylvania).

The acid then used, and that which still predominates today, was muriatic or hydrochloric acid. Its use was severely restricted, however, until the 1930s because of its extremely corrosive effect on casings and tubulars. In 1932 the Dow Chemical Company and the Pure Oil Company introduced their method of acidizing. The former company had developed an additive for the acid which deposited a molecular layer on any metal contacted and thus protected it from corrosion by the acid. The latter company had perfected and provided the know-how to apply this inhibited acid to the desired pay horizon.

Since then, many refinements to the technique and many more additives to modify the properties of the acid (its reaction rate, its tendency to form stable emulsions with formation water, etc.) have been developed. So much so that it is conservatively estimated that some 200,000,000 gallons of acid were used during 1957 in more than 80,000 wells in the Western Hemisphere alone.*

Despite all this acitivity over the past 25 years, little more is known now about the nature of the acidizing process than that postulated by Frasch and Van Dyke more than 60 years ago, *i.e.* hydrochloric acid dissolves limestone, hence there will be a local increase in porosity and permeability around the well bore will result in increased production. And yet, despite all of this fundamental knowledge, huge sums of money have been expended and are still expended in drilling wildcats into limestone formations. This has been done with confidence, in the strong belief that if an oil-bearing stratum is discovered, it can, regardless of physical characteristics, be made commercially profitable by the use of acid. Indeed, so great is this faith in acidizing that, in some fields, all wells are automatically acidized on completion and before testing, and often this is done without reference to economic considerations involved.

Of course, during this time, a wealth of field experience

* Fitzgerald, P. E. *Petroleum, Lond.*, 1959, **22** (5), 179–80.

and information has been amassed. Certain techniques have been adopted for specific areas, and these techniques are often followed blindly, somewhat as one roots for one's team—win or lose. There are those oilmen who will intuitively wash or squeeze, single or multi-stage acid treat, overall or selectively treat a limestone oil-producing horizon simply on a prejudicial basis that they had applied the technique with success previously. Often the results obtained are very outstanding, but the point I wish to make is that we have no assurance that even though the results might be good or moderate, that those results obtained are the best that could have been achieved, and with the least capital expenditure, unless we have a yardstick with which to judge. A well-established and thoroughly understood theory describing the acidizing process could well serve as such a yardstick.

It is here, I believe, that the value of the paper presented this evening will be found. There are places in the world, as we all know, where the supply of large amounts of acid is both difficult and expensive. It seems highly likely that as the years go by there will be future and great discoveries of limestone formations, and no doubt many of these formations will be inherently difficult or uneconomic to produce, unless properly programmed and developed. In cases such as these, it seems to me that the proper and economical use of acid is essential to a successful development programme.

Tonight we have been told that, in limestones of the nature described, the most economical use of acid is, as I understand it, to let the acid find its easiest path, which will be into the larger pore channels, because there its effect in improving production capacity of the well will be greatest.

The proving and expansion of this theory into practice could well result in greater efficiency and tremendous economies within the industry. This theory tends to under-emphasize the improvement of so-called " tight " zones by the use of " ball-sealers " and other rather complicated and expensive down-the-hole specialized techniques, and to place the emphasis on more simple acid squeeze jobs covering an entire interval. If this marks the return to the simple life, we would do well to applaud the results of Dr Rowan's technical study.

Dr Rowan suggests that further work should be done to determine that portion of the pore size distribution which may be classed as " effective." I am wondering whether, since preparing this paper last December, he has pursued this matter further, and, if so, whether he has been able to deduce yet further conclusions from such studies.

Dr G. Rowan: In reply to Mr Holley's question regarding the portion of the pore size distribution which may be classed as " effective," some work is at present being done along these lines, but, as yet, no definite conclusions may be drawn. The main problem under active consideration is the extension of this theory to cover practical field cases with the object of discovering what should be the radial permeability variation around the well bore. Using a normal, *i.e.* unretarded, acid the permeability of the acidized zone would almost certainly not be uniform. As the increase in well productivity is a function of the permeability variation around the well bore, it is essential to know the form of this variation in order to calculate the most economical quantity of acid to inject for any desired degree of improvement.

W. J. Baker: I would like to ask Dr Rowan if he could tell us anything on the relative merits of using a given quantity of acid in two different ways. Shall we say we have 10,000 gallons of acid, which we propose to use? Would it be better to put it all in at once, or to divide it into ten portions, and put it in with ten separate shots, the difference being, of course, that in the first case one obtains far deeper radial penetration into the formation, and in the second case, one would be getting a far bigger increase of permeability in the more immediate vicinity of the well bore.

Dr G. Rowan: From some preliminary calculations which have been made, it would appear that it is preferable to subdivide the acid and use a multiple treatment rather than to aim at a deep penetration with a single treatment. The steady-state production increase depends upon two factors, the permeability around the well bore and the radius of penetration. To a certain extent one of these factors may be adjusted at the expense of the other to give the same ultimate effect. With a given quantity of acid, however, a higher permeability in a limited zone around the well bore seems to produce the more favourable result, and this would be obtained by multiple injection. Naturally these calculations refer to a limestone of the type previously discussed; with a fissured system the state of affairs may be somewhat different.

A. Beeby-Thompson: After listening to an interesting but highly mathematical paper I was led to wonder whether the deductions could be related with limestone formations which did not rely upon effusion from a rock face. From experience of many years I came to the conclusion that all major limestone fields obtained the bulk of production from fissured or cellular zones. I remember discussing this aspect with Dr Lees many years ago, before it was discovered how extensive were the fissure systems in the Persian fields. He then thought that oil issued from the many hair-like cracks in the limestone. Now we know in actual fact most of the great limestone fields of Iraq, Iran, and Mexico drew their oil from fissures rather than vesicules. Even in the very early shallow fields of the Alleghanies, where the original Drake well was sunk, fortunately-placed wells yielded at a depth of a few hundred feet as much as 5000–10,000 bd. Obviously the few hundred feet of limestone penetrated could not have given this rate of flow by mere effusion from a crystalline rock face. In the very productive limestone fields of Iran and Iraq, interference of wells separated by miles has clearly indicated the importance of rock fissures on production capacity.

I am led to wonder whether without cracking the tight limestones by explosives or other means any large or sustained productions would result. In the old days there was always the uncertainty as to the relative merits of acidizing and shattering of hard limestones by explosives. Perhaps the author could say something on the relative value of acidizing and shooting in tight calcareous formations.

Dr G. Rowan: In a given type of formation the object of a production stimulation technique is to reduce the pressure drop in the region around the well bore. This results in greater production for a given draw-down. The use of high explosive to produce a limited fracture zone around the well bore may result in a greater initial increase in production than would be produced by acid treatment. In a tight limestone one might expect the effect to decline fairly rapidly, with the result that the long term benefits attained by " shooting " and acidizing might not differ significantly.

H. C. Lack: I would like to ask Dr Rowan whether, in those formulæ which he has deduced and the consequent new doctrine which he has propounded, there is anything to tell us whether, when acidizing a well, we should allow it to stand with its acid in it, or whether, as soon as we have pumped all the acid, we should get it out again? In other words, is there any advantage in allowing a well to stand with acid in it, as compared with the common practice of " rocking " the acid into and out of the formation?

Dr G. Rowan: I would suggest that the acid should only be allowed to remain in the formation long enough for the reaction to take place. After this time it would seem desirable to get rid of the acid as soon as possible. An estimate of the reaction time for any particular formation could be obtained from laboratory tests.

W. J. Baker: It is very desirable, in my experience, not to allow the acid to remain still at all, because there is always a certain amount of insoluble débris from the reaction. If it is kept moving, one is much more likely to keep it from settling out in the pore channels, and possibly blocking them. In fact, I think it has been found in the laboratory that one can get a complete blockage in a core; even though the acid makes an enormous hole to start with one finishes up with it completely blocked.

Dr G. Rowan: Yes, that is quite true. We did find that, even with cores of the type shown, on cutting the core along one of the large holes a considerable amount of plugging material was present. This, we were informed by the geologists, was composed of a certain amount of shell débris which had a different reaction rate from the bulk of the limestone. The shell débris, together with some emulsion formed by the acid and the particular type of crude, formed a fairly effective plugging material. It was rather surprising to find that, even with a hole of the order of $\frac{1}{2}$ inch in diameter, the permeability of the core was only of the order of 100 mD. In view of these observations I would agree with Mr Baker that to keep the acid moving at all times may help to prevent the formation of such plugs.

M. J. Jeffries-Harris: Would Dr Rowan please give his opinions as to the correct injection pressure of the acid into the formation to be treated?

Should it be pumped at a pressure greater than the parting of hydraulic fracturing pressure; or should it be controlled so as to move through the natural porous structure, at a rate and injection pressure relative to the normal, non-artificially-fractured, permeability?

Dr G. Rowan: I would suggest that, for acid treatment, the injection pressure should be related to the normal formation permeability. Injection at pressures sufficient to induce fractures is somewhat beyond the scope of the present theory.

R. J. Holley: I would like to add two comments. One is that I was very pleased to read recently in the literature that Dr Muskett, although in general terms, completely supported Dr Rowan's theory. Secondly, on this question of these complicated techniques of trying to acidize the tighter formations, there are other reasons, I believe, for having gone to that extremity, although it has got out of hand. I know, for example, that there are some operators who have tried this method of letting the acid go into the larger channels, but unfortunately all areas are not blessed as is the Middle East, and they would be dealing with, let us say, 12 or 15 feet of limestone. The net result is that they got the porosity, but they also got the water, and I think in many cases that led to this business of, " Well, now, let's not get the biggest porosity; let us get this very little tight vein." I think it is from some of those necessities that many of these more complicated and expensive techniques have developed.

S. Howe: I wonder if we have the correct impression of the limestone under study? As I understand it, Dr Rowan has dealt with an earthy, almost oolitic, porous type of limestone. On production, would it not behave more like a sandstone reservoir than a classical type of limestone field in which the rock matrix is virtually impervious and the productivity is dependent almost entirely on an extensive intercommunicating fracture system?

Dr Rowan has postulated that, in the type of limestone considered, the greatest permeability increase is to be achieved by letting the acid find its own path into the formation. However, this maximum permeability increase per unit of acid consumed is largely localized in the larger pore channels. One would, therefore, not only be increasing the permeability around the well bore, but also increasing the permeability variation in the well. In some instances, for example, in a well intended for fluid injection, one might wish not only to increase the permeability but also to reduce the permeability variation. Would it not be better to aim the acid at the tighter zones in such cases?

Dr G. Rowan: With regard to the first point, the limestone considered in this study was not of the porous, oolitic variety, but rather of the massive type. The work of Nougaro and Labbé, however, was concerned with the porous type of limestone possessing an approximately uniform pore structure.

To answer the second point it is necessary to differentiate between the application of acid treatment to primary and to secondary recovery. In the great majority of cases, acid treatment is used to stimulate primary production with the object of attaining the maximum possible well productivity. This would be expected to be given by the maximum overall increase in permeability around the well bore. The theory indicates that this should be obtained by allowing the acid to penetrate into the formation without restriction. Naturally the permeability variation in the well will be increased by this method, but, in the case of primary production, this should not constitute any great disadvantage.

In the case of an injection well, as Mr Howe points out, it may be desirable not to increase the permeability variation. For such wells, selective acid treatment might have to be considered.

W. J. Baker: I think the basic idea of this selective acid treatment is to make sure that one drains all the limestone. If one is just going to increase the permeability in the best part, one is draining that more or less to the exclusion of the rest of it. I do not think that is strictly true, unless one has continuous shale partings, or something of that sort, because by taking the major part of the production from the most permeable layers one will set up a differential between those layers and the others, and the less permeable layers will drain into the more permeable ones. I therefore think that the suggestion that selective acidization is unnecessary is correct, unless one actually has completely impermeable layers, such as a continuous shale parting in the middle of the limestone.

The Chairman then called on **T. Dewhurst** to propose a vote of thanks, which was carried with acclamation.

A RAPID METHOD OF DISTILLATION FOR THE ROUTINE QUALITY CONTROL OF REFINERY PRODUCTS *

By W. H. TOPHAM †

SUMMARY

A rapid method of distillation is described for the routine volatility control of refinery products where a single point on the distillation curve is sufficient information. Repeatability is comparable with the standard methods.

ANALYSES of the distribution of testing effort in a refinery control laboratory have shown that about 10 per cent of the total work load is devoted to the distillation of unit products for routine plant control purposes. A single full distillation (IP 123) takes about 30 to 45 minutes to complete, although an experienced operator with suitable grouping of apparatus can carry out simultaneously four plant control distillations (noting only the IBP, 10 per cent, 50 per cent, and 90 per cent evaporated temperatures and the FBP) and complete these four within the hour. The use of commercial instruments which simulate automatically the standard methods achieves no reduction in testing time, and the capital expenditure involved may be considerable.

Experience has shown that the value for a single point on the distillation curve (usually the 90 per cent recovered temperature) is often sufficient information for the routine volatility control of many refinery products, and since the standard methods are not mandatory for control purposes, their replacement by a rapid procedure offered significant savings in time and manpower. A rapid automatic method of distillation has been developed on this basis. With this rapid method determinations take less than 10 minutes (only 2 minutes operator time), repeatability is comparable with the standard methods, and the results obtained over the range 50 to 90 per cent recovered are sufficiently close to those obtained by the standard methods to be of direct use for many plant control purposes without correlation.

The determination of a single point on the distillation curve can be approached in two ways:

(1) the temperature can be recorded at which a given volume of distillate has collected in the receiver, or

(2) the volume of distillate collected at a predetermined temperature can be measured.

The second method was chosen for development because of the simplicity of the apparatus and the ease with which it could be adapted for automatic operation using inexpensive commercially available components.

The basic requirements of the apparatus were defined as follows:

(*a*) a small capacity flask and an efficient condenser having very small sample hold-up;

(*b*) a heater able to produce rapid boiling but of low heat capacity;

(*c*) temperature measurement by a thermocouple, which could be accurately and reproducibly located in the neck of the flask;

(*d*) a temperature indicator with cut-out device operable at any temperature within the range of the instrument.

A detailed description of the apparatus is given in the appendix.

Repeatability

The 90 per cent recovery points were determined by different observers on samples of gasoline, kerosine, and gas oil over a period of five weeks. The temperature selector was reset before each distillation. From the results shown in Table I it can be seen that

TABLE I

Repeatability of the Rapid Method

Sample type	Motor gasoline	Kerosine	Gas oil
Cut point, °C	168·5	228·5	324
No. of determinations	14	14	14
Mean recovery, % vol	90·0	90·0	90·0
Long-term repeatability of the rapid method, % vol	1·7	2·0	1·8
IP repeatability, % vol	2	2	2
IP reproducibility, % vol	3	3	3

the long-term repeatability figures compare favourably with the precision data quoted in the IP Methods of Test for distillation.

Correlation with IP Standard Method

This was of considerable importance, as control of refinery distillation equipment is traditionally based on IP results and, since the rapid method is not applicable to all samples, reporting of results in terms

* MS received 14 July 1959.

† BP Refinery (Kent) Ltd.

of two test methods could lead to confusion. Hence a conversion of rapid results to IP is necessary.

Distillations were carried out on a wide range of refinery products, using both the rapid method and the standard method (IP 123) in order to assess the correlation between them. In most cases cut point temperatures were chosen to give approximately 90 per cent recovery, as this was of greatest interest.

of equivalent IP recoveries by the rapid method would be practicable only after extensive correlation work. Fortunately, recoveries other than at the 90 per cent level are seldom used for plant control purposes in the refinery.

Three instruments of the type described (manufactured under licence by F. J. Hone & Co. Ltd) have now been in regular use since August 1958 in this

TABLE II

Correlation of Rapid Method with IP

Sample No.	Type	Heater, volts	IP distillation		Cut Pt °C	Per cent recovery at cut point		90% recovered temp	
			IBP	FBP		Rapid [1]	IP [1] method	Rapid [2]	IP method
1	Gasoline	15	36	71	51	91	83	50	55
2	Cracked spirit	19	34	98	80	91	90	78·5	80
3	S.R. benzine	24	78	146	100	49	42·5	—	—
					110	67	65	—	—
					126	90·5	89	125·5	126·5
4	S.R. benzine	27	87	157	105	25	18	—	—
					110	56	52·5	—	—
					130	74	71·5	—	—
					142	90	89·5	142	142·5
5	Naphtha	30	136	172	162	89·5	93·5	162	160
6	Naphtha	33	148	182	171	88	91	172	170
7	Cracked spirit	30	69	219	130	45	38·5	—	—
					155	74·5	71	—	—
					180	92	90·5	176	179
8	Kerosine	38	171	253	245	90·5	94	244·5	240
9	Kerosine	38	180	261	245	90	90·5	245	244
10	Gas oil	33	185	328	309	89·5	90	310	309

[1] Results quoted for both methods are mean values of a number of determinations on each sample.
[2] The "equivalent 90% recovered temperature" is calculated from the per cent recovery at the cut point temperature and the slope of the IP distillation curve.

From the results shown in Table II it can be seen that, with the exception of gasoline, correlation of the two methods at the 90 per cent recovered level is sufficiently close for determination of IP 90 per cent temperatures for many refinery control purposes to be carried out by the rapid method without the necessity of correlation work. Where closer correlation is required small corrections can be applied on the basis of experience.

In this laboratory simple tables have been prepared showing the IP 90 per cent temperatures corresponding to the per cent recoveries at a selected temperature for a range of products.

Below the 50 per cent recovered level the divergence of the two methods is considerable and the assessment

laboratory for the determination of 90 per cent temperatures on samples of benzine, naphtha, kerosine, and light and heavy cracked spirits for the routine control of three refinery distillation columns. Results are available in a fraction of the time previously taken, a factor of considerable importance when changing unit operating conditions, or during periods of upset.

ACKNOWLEDGMENTS

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APPENDIX

Description of the Apparatus

The apparatus * is shown in Figs 1 and 2. A 25-ml distillation flask with B.14 ground glass socket at the neck is attached

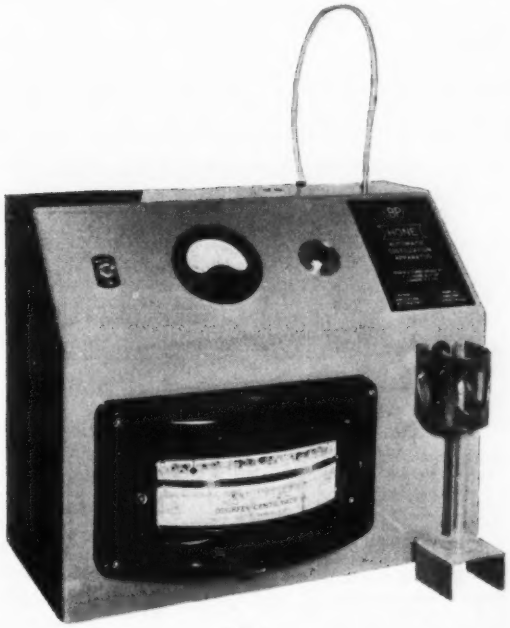


FIG 1

to a short water-cooled metal condenser using a glass to metal seal. For high temperature distillations (*e.g.* gas oil) the flask is lagged with a removable asbestos fibre jacket. The 18 SWG iron-constantan thermocouple is sealed through a ridged PTFE stopper with B.14 taper ensuring reproducibility in positioning the thermocouple and giving a vapour-tight seal without lubrication. The thermocouple is connected to a "Transitrol" Type 990 indicating temperature controller (Ether Ltd). This moving coil instrument incorporates a photo-transistor relay circuit which is used to switch off the flask heater at a preselected temperature within the range 0°–320° C.

The heater consists of 32 turns (15 ohms) of "Brightray" resistance tape wound on a 2-inch × 1-inch mica former supported on two upright copper wires which also serve as power leads. The heater voltage is provided by a "Variac" transformer and measured on a panel-mounted voltmeter. The whole assembly is mounted inside a sheet steel cabinet 16 inches × 14 inches × 8 inches. Distillate is collected in a 10-ml Grade A graduated cylinder.

Method of Operation

10 ml of the sample is introduced into the flask from a pipette (Grade A). The thermocouple is positioned in the neck of the flask and the distillate receiver placed beneath the condenser spout. The control arm pointer on the temperature indicator is set to the required cut-out temperature, the heater is switched on by means of the press-button on the control panel, and the voltage set to give 90 per cent recovery in approximately 7 to 8 minutes. Typical heater voltages are included in Table II.

Distillation proceeds until the temperature at the thermocouple reaches the selected cut-out temperature, at which point the heater is automatically switched off and distillation ceases. After allowing half a minute for draining (approximately 0·2 ml is collected after heater cut-off) the volume of distillate is noted to the nearest 0·05 ml and expressed as a percentage of the original sample volume.

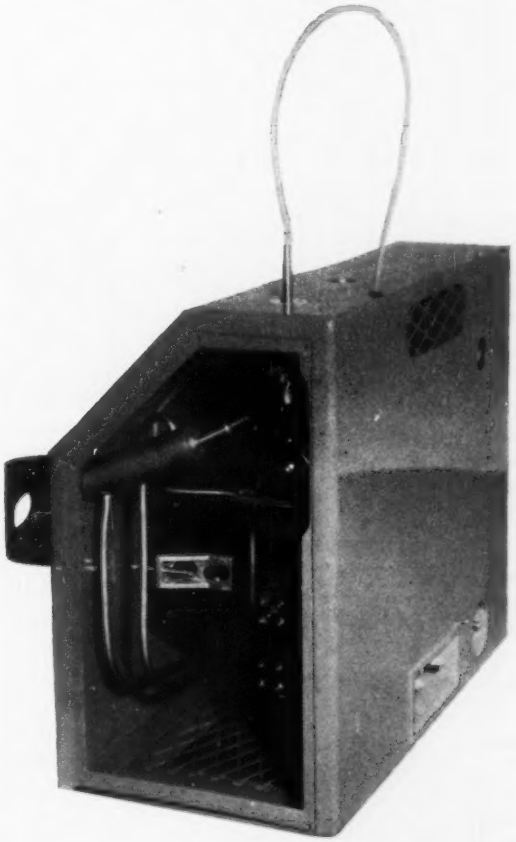


FIG 2

After each distillation the contents of the flask and receiver are removed by suction, for which purpose a length of fine-bore nylon tube has proved very useful. Rinsing and drying of the apparatus is not necessary. For repetitive work on similar samples the cut-out temperature and heater voltage need no alteration.

* Patent applied for.

LOAD-CARRYING CAPACITY TEST FOR OILS IAE GEAR MACHINE *

IP 166/60 Tentative

Introduction

1. At a given speed the load which can be transmitted by a pair of spur or helical gears is different for different lubricants, *i.e.* the lubricants have different load-carrying capacities. If the load-carrying capacity of a lubricant is exceeded it fails to protect the gears and the surfaces of the gear teeth become damaged. Some of the forms of damage which can occur are described in Appendix I. In the IAE gear machine, which is used to determine the relative load-carrying capacity of lubricants, the most common form of damage is scuffing, although scoring is also experienced. As the incidence of both of these types of failure is affected by the lubricant they are used as criteria of performance in the test described below.

Scope

2. This method may be used to assess the relative load-carrying capacities of oils when used to lubricate steel/steel spur gears. The results are generally assumed to be applicable also to steel/steel helical gears.

Outline of Method

3. A special gear rig is operated for a series of short periods, at increasing loads, the oil under test being used to lubricate the test gears. The performance of the oil is evaluated by the load at which scuffing or scoring of the gear surfaces occurs.

Apparatus

4. (*a*) *Test Rig*—A power circulating gear test rig of the type developed by the Institution of Automobile Engineers employing special test gears. It is known as the IAE 3¼-inch Centres Gear Lubricant Testing Machine (IAE Gear Machine). The rig is described in Appendix B1.

(*b*) *Test Gears*—as described in Appendix II, B2.

Materials

5. *White Spirit*—conforming to BS 245 : 1956.

Preparation of Apparatus

6. (*a*) Follow the makers' "Operating and Servicing Instructions" for the general operation of the machine.

(*b*) Before commencing a test on a new lubricant, remove and discard the paper filter element and flush the test oil system through twice for 15 min, first with the white spirit at room temperature and then with the test lubricant at the test temperature (Table I). Switch off the heater, allow the oil to drain for 15 min, during which time insert a new paper filter-element and refill with 1 IG of the test lubricant.

(*c*) When repeating tests on the same lubricant the oil must be changed, but the flushing procedure may be omitted. Change the filter-element for every other test.

(*d*) Thoroughly cleanse the gears in the white spirit before fitting them in the machine and immediately prior to the test.

Procedure

7. (*a*) The three sets of nominal conditions to be used for scuffing tests are given in Table I.

TABLE I

Normal conditions	A	B	C
Pinion speed, rev/min	2000	4000	6000
Test oil temperature, °C	60	70	110
Test oil temperature, °F	140	158	230
Test oil flow, pt/min	½	1	1
Initial load (18-inch lever arm), lb	10	10	10
Load increment, lb	5	5	5
Test period, min	5	5	5
Rest period (time between tests), min	5	5	5

(*b*) Fit a clean matched pair of test gears, which must bear the same serial number.

(*c*) Switch on the heater and test oil pump and allow the oil to circulate until the temperature remains constant at the selected value (Table I). Then adjust the test oil flow to the correct rate.

(*d*) Apply an initial load of 10 lb by the application of the standard 18-inch lever arm and weight carrier without added weights.

(*e*) Operate the machine under the appropriate conditions for 5 min (Table I).

(*f*) Stop the machine and when it comes to rest switch off the test oil pump. Allow the machine to remain at rest for 5 min, during which period examine the teeth of the test gears.

(*g*) Visual inspection will show either one of the conditions of gear teeth given in Appendix I, or that no change in the appearance of the gear teeth has occurred.

(*h*) If no scuffing or scoring has occurred increase

* This methods replace IP 166/58T published in the 1959 edition of Standard Methods under the title "Scuffing Test of Gear Oil." The revised method defines more exactly the degree of scuffing or scoring constituting failure. A section on Precision and an Appendix defining the various forms of tooth damage which may occur during a test are included.

the lever load by the addition of a 5-lb weight. To avoid shock loads the lever arm and weights must be applied to the coupling before the clamp bolts are slackened. One minute before starting the machine switch on the test oil pump.

(*i*) Operate the machine for a further 5 min and repeat the procedure from (*f*).

(*j*) Repeat the 5-min operation and the 5-min rest period with increasing loads in increments of 5 lb until the teeth are found to be scuffed or scored to the extent described in Section 8.

(*k*) The onset of scuffing may be indicated by the following:

(i) Change in note of the noise of the rig;

(ii) Appearance of smoke in the test box;

(iii) A sudden increase in the current taken by the driving motor;

(iv) A sudden increase in the temperature of the oil emerging from the contact zone (as indicated by the thermocouple, if this is fitted).

In such cases the rig shall be stopped immediately in order to avoid the possibility of excessive damage to the gears occurring. It must be emphasized, however, that these changes are not always coincident with the onset of scuffing, and the criterion in every case is the actual occurrence of scuffing as detected visually.

The onset of scoring is less pronounced and will usually be noticed only when the machine is stopped for inspection of the gears.

(*l*) If the machine has been stopped before the end of the 5-min period and complete failure (see Section 8) has not occurred, restart the machine immediately and complete the 5-min run continuing the test according to paras. (*h*), (*i*), (*j*), (*m*), (*n*).

(*m*) If on inspection initial failure (see Section 8) is found to have occurred, record the load as the Initial Failure load and continue the test until failure is complete as illustrated in Figs 1 and 2.

(*n*) If on inspection failure is complete (see Section 8) record the load as the Complete Failure load.

(*o*) If the difference between the Initial and Complete Failure loads exceeds four load increments (20 lb), reject the result and repeat the test.

(*p*) Carry out a total of four tests under the chosen set of conditions (Table I) using one face of each of four pairs of gears on any one lubricant.

Extent of Damage

8. When scuffing occurs (as defined in Appendix I, A1, (*c*) and illustrated in Fig 1) the damage area extends across the tooth face and from the tip of the tooth down towards the pitch line. Usually the region around the pitch line is undamaged, the extent of the undamaged area varying with the load and duration at the failure condition. The mating tooth scuffs over the corresponding mating area (*i.e.* flank).

FIG 1
TYPICAL SCUFFED TOOTH

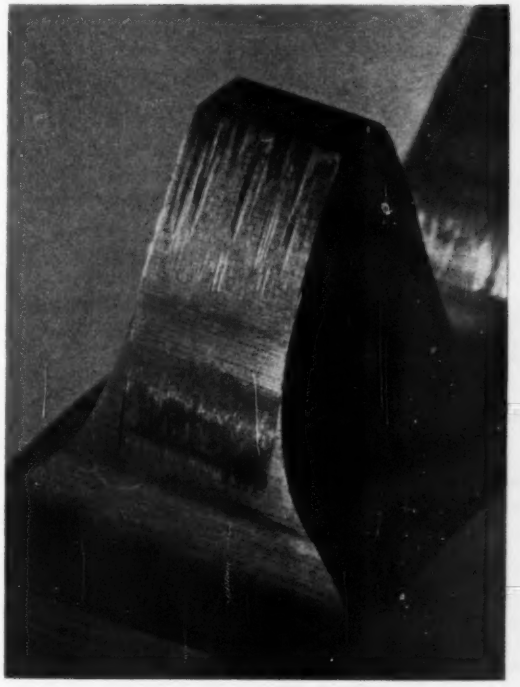


FIG 2
TYPICAL SCORED TOOTH

When scoring occurs (as defined in Appendix I, A1, (*d*) and illustrated in Fig 2) the extent of surface damage may vary from a small area of the tooth up to as much as is observed for scuffing. The mating tooth generally scores over the corresponding mating area.

Failure shall be taken as the point at which at least 60 per cent of the face area (Fig 3) is scuffed

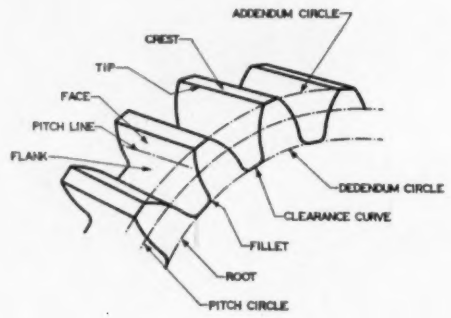


FIG 3

GEAR TOOTH NOMENCLATURE

and/or scored. If only one face/flank (Fig 3) combination is scuffed or scored the load at which it occurs shall be termed the Initial Failure load. If both face/flank combinations are scuffed and/or scored this shall be termed the Complete Failure load.

Calculation and Reporting

9. Report the Initial and Complete Failure loads for each of the four tests. When failure is complete at one load report this as both the Initial and Complete Failure loads.

From the eight results obtained, calculate the arithmetic mean to the nearest whole number.

Report the eight results and their mean as the Load-Carrying Capacity, IP 166, condition A, B, or C (Table I) as appropriate.

Precision

Duplicate results, each being the mean of the eight failure loads obtained in carrying out the test, should not differ by more than the following amounts:

Repeatability	Reproducibility
$5 \text{ lb} + 0{\cdot}07x$	$16 \text{ lb} + 0{\cdot}21x$

where x is the mean of the duplicate results.

NOTE 1: These precision values have been obtained by statistical examination (see Appendix E) of inter-laboratory test results using the IP 166/58T test procedure. It is believed, however, that they offer a suitable guide to the precision of this method also.

APPENDIX I

TYPES OF SURFACE DAMAGE

A1. When the load on a pair of spur gears is progressively increased the appearance of the contact surfaces alters. Not all of the types of damage listed below may occur in any one test but their incidence should be recorded. Definitions of surface damage for the purposes of this test are:

(*a*) *Scratching.* Shallow lines, usually running perpendicular to the line of contact. The lines frequently do not extend all the way from tip to root.

(*b*) *Polishing.* A smoothing over of the grinding marks with no tearing or grooving of the surface. With certain types of oils this may be so pronounced that the IP Failure load cannot be determined.

(*c*) *Scuffing.* Roughening of the surface by welding and tearing as shown in Fig 1. This condition usually appears suddenly.

(*d*) *Scoring.* Grooving of the surface in the direction of sliding, accompanied by partial removal of the grinding marks, with no scuffing present. This condition is shown in Fig 2. It is progressive and gets worse as the load is increased.

(*e*) *Edge flaking.* The removal of large particles of metal from the edge of the tooth. This condition frequently occurs if a gear is progressively loaded beyond the point at which scoring is extensive.

(*f*) *Pitting.* Small irregular cavities in the tooth surface brought about by the breaking out of surface metal. This condition is indicative of surface overloading.

(*g*) *Tooth breakage.* The loss of part or the whole of one or more teeth. This condition may occur as a result of continuing to run pitted gears but may also occur at very high loads without any other sign of surface distress.

When carrying out a test only scuffing (*c*) and scoring (*d*) should be considered as criteria for lubricant failure, but other surface changes should be recorded as they occur.

APPENDIX II

APPARATUS

B1. (*a*) *Gear Test Rig*—a power-circulating type gear test rig of the type developed by the Institution of Automobile Engineers employing special test gears. It is known as the IAE 3¼-inch Centres Gear Lubricant Testing Machine. The sole authorized manufacturer of the rig is W. E. Sykes Ltd, Staines, Middlesex, England. The rig consists of two gear boxes, containing respectively the test gear and the "power return" gears, coupled in parallel by two torsion shafts, one of which carries a split coupling so that torque can be applied to the system. Loads up to 200 lb may be applied on the loading lever, corresponding to 300 lb/ft torque and approximately 12,000 lb/inch tooth face pitch-line loading. The apparatus shall have separate oil systems for the test gears and the power return gears. The test oil system shall include means of controlling both the flow and temperature of the oil, heating being carried out by indirect means. Both magnetic and paper element filters shall be fitted. The use of copper base metals shall be avoided as far as possible. The temperature of the oil shall be controlled by a bulb type indicator controller, the sensitive element of which shall be located immediately prior to the oil jet.

Serial Numbers of the machines produced to date are:

Mark I Machine S 537/1 to S 537/9

(The initial letter S may or may not be present on certain machines)

Mark II Machine S 2747/1 to S 2747/12

Mark III Machine MP 131/1 to MP 131/6

(*b*) *Magnetic Filter*—a Phillips Type E 7715 filter.

(*c*) *Paper Element Filter*—a Purolator "Micronic" MF 2604 filter fitted with an MF–HA26 element.

(*d*) *Oil Feed Jet*—the gears shall be fed with oil by an 0·8 mm dia jet (Sykes Drg No. AM 37077) directed vertically downwards towards the meshing point of the gears.

(*e*) *Test Constants*—if the pinion rev/min is n and the load on the standard 18-inch lever arm is W lb, then for the standard 15/16 ratio gears the following apply:

Pitch-line velocity, ft/min	$= 0{\cdot}82\ n$
Maximum sliding velocity, ft/min	$= 0{\cdot}39\ n$
Pitch-line tooth loading lb/inch	$= 61{\cdot}0\ W$
Horse power	$= 0{\cdot}00029\ W\ n$

B2. *Test Gears*—brief details are given in Table II and full details in Sykes Drg Nos. AM 13678G, AM 13677G, and BM 13889G. Authorized suppliers are W. E. Sykes Ltd, Staines, Middlesex, England, and British Gear Grinding and Manufacturing Company Ltd, Standard Road, Park Royal, London, N.W.10, England.

TABLE II

Details of Test Gears

Number of teeth	15	16
Working pitch-circle dia, inches	3·145	3·355
Base circle dia, inches	2·819	3·007
Working pressure angle	26° 19′	26° 19′
Diametral pitch	4·769	4·769
Face width, inch	0·188 ± 0·001	0·188 ± 0·001
Tip relief, inch	0·0003 ± 0·0001	0·0005 ± 0·0001

The gears shall be made of 2 per cent nickel–molybdenum steel conforming to BS971: 1950 (EN 34). Either face may be used for the test. Used gears must not be reground and used for further tests.

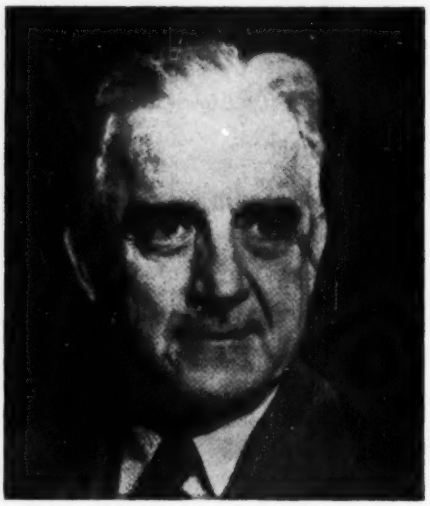
OBITUARY

REGINALD KILLMASTER STRATFORD

1895–1959

It is with great regret that we record the death in London on 20 April 1959 of Dr R. K. Stratford—a man distinguished not only for his achievements in the fields of chemistry and petroleum but also for his interests in research, in education, and in public affairs. In many ways a pioneer, he exerted an influence which has lasted and will continue to last in the minds of those fortunate enough to know him.

R. K. Stratford was born on 10 August 1895 at Brantford, Ontario, the tenth child of a family with twelve children. In boyhood he suffered severe illness which he overcame as much by force of character as by any other means. It was during this period that he acquired, by reading, much of his early interest in a wide variety of subjects. To this he added later an expert knowledge of gardening and horticulture. In 1915 he obtained his first degree, that of Bachelor of Scientific Agriculture, at the Ontario Agricultural College and after a few years as an agricultural representative, and carrying out post-graduate work and demonstrating, gained the M.Sc. at Amherst Agricultural College. Obtaining a scholarship, Stratford worked from 1922 to 1924 at the Université de Lyon, being awarded the D.Sc. degree in the latter year. At Lyon he worked with Victor Grignard and in 1924 published his first paper on "The Catalytic Decomposition of Hexahydro-Aromatic and Saturated Aliphatic Hydrocarbons," followed in 1925 and 1929 by two important papers on the action of aluminium chloride on hydrocarbons. This work, published in the *Comptes Rendues* and other French journals, constituted Stratford's introduction to hydrocarbon chemistry. The period also engendered in him a lasting affection for France and the French people.



Returning to Canada in 1924, now married and with a desire to teach, Stratford found posts difficult to obtain. For this reason he turned his attention to industry and, probably on account of his work on hydrocarbons in France, joined the Imperial Oil Company at Sarnia, the company having decided to carry out some of its own research work. At that time the employment of highly qualified scientists in industry was not at all widespread and it was suggested to Stratford that he had too many degrees and was probably too academic in his outlook! From the one-man research department which he started with little equipment or facilities, Dr Stratford built up a small but thriving organization and in 1929 was appointed Director of Research. He continued in this position as head of a steadily expanding research laboratory until 1951, when he became scientific adviser to Imperial Oil Company.

Stratford entered the petroleum industry at a time when the scientific background of petroleum refining and utilization had been very little developed, when chemists were largely employed in routine and control work, and laboratory equipment was crude and primitive by to-day's standards. The change in outlook was beginning, however, and the next ten years saw great changes in the industry.

Stratford's own contributions to this change were noteworthy and included the development of treating methods, such as the countercurrent clay process and, starting in the period 1928–30, the introduction of the widely used phenol-treating process for the upgrading of lubricating oils by extraction. Later work resulted in the development of the "Suspensoid" cracking process which was used at Sarnia for some years before the introduction of the fluidized catalytic cracking process; he and his colleagues also worked on dewaxing processes, on fundamental studies of asphalts and grease structure, and innumerable other subjects. During his career he published some 50 papers and patents.

Dr Stratford's versatility showed itself not only in his work in the petroleum industry but in many other activities. He was an expert and enthusiastic gardener with a large and very beautiful garden on the banks of the St Clair River near Sarnia. He was actively interested in amateur theatricals and served as president, director, or producer of the Sarnia Drama

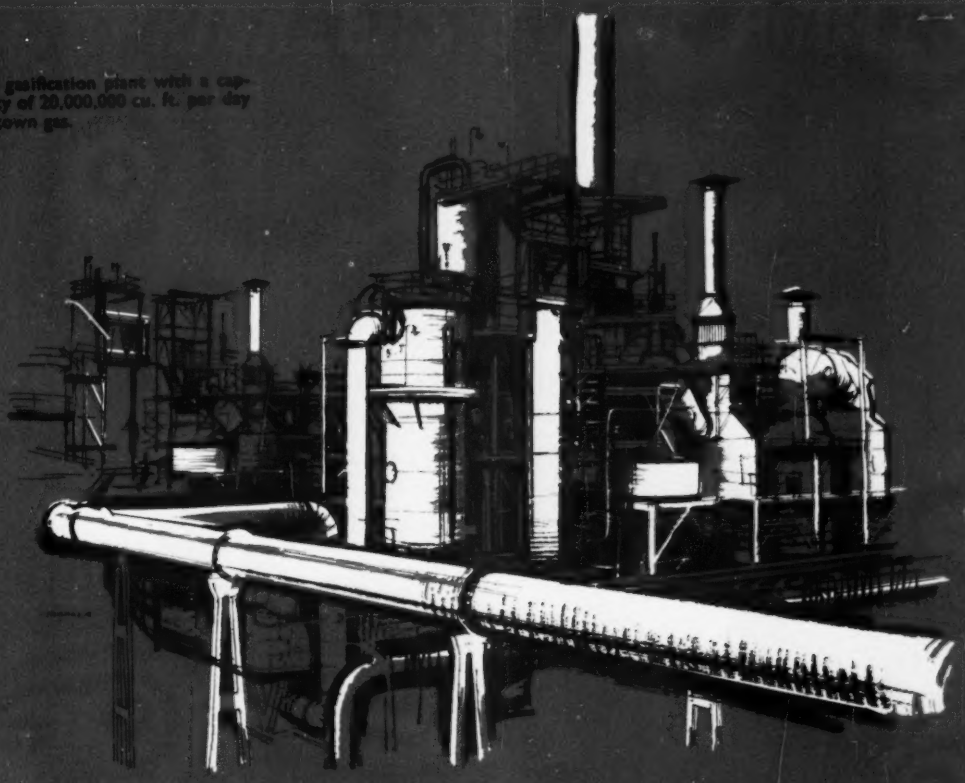
League for many years, and was knowledgeable on both music and literature. Other local public services included the chairmanship of Welfare Committees, Town Planning Commissions, and public school boards. On the professional and research side he was an active member of many scientific bodies; he was a Fellow of the Institute of Petroleum, which he joined in 1934, served as President of the Canadian Institute of Chemistry and Chairman of the Chemical Institute of Canada and of the Canadian Section of the Society of Chemical Industry and as President of the Research Council of Ontario, was a member of the Ontario Cancer Treatment and Research Foundation, and for some years was the Canadian member of the Permanent Council of the World Petroleum Congress.

Dr Stratford was the recipient of many awards and honours in recognition of his achievements and services both to science and to the community. The University of Western Ontario conferred on him an honorary D.Sc. in 1949 and Queens University an honorary LL.D. in 1953. In 1954 he was presented with the Medal of the Canadian Section of the Society of Chemical Industry.

Few men have had a fuller or more productive life than R. K. Stratford. His wide interests, ability, and versatility, soundness of judgment and foresight combined with conversational powers, an interest in his fellow men, and an easy friendship combined to make a man who has left a lasting impression on both petroleum science and in Canadian life. A man of strong opinions, he had the gift of expressing them in a manner which was, at the same time, both forceful and charming.

His passing will be felt deeply by his friends in many countries and all will remember him with real affection and extend their sympathy to Mrs Stratford and her family in their loss.

E. B. E.



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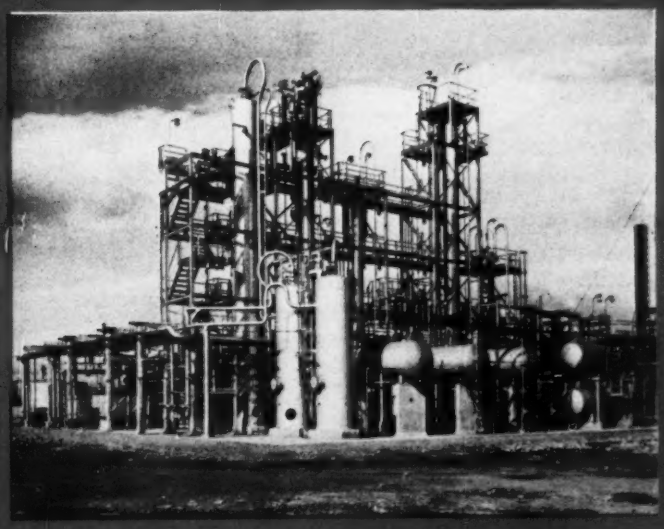

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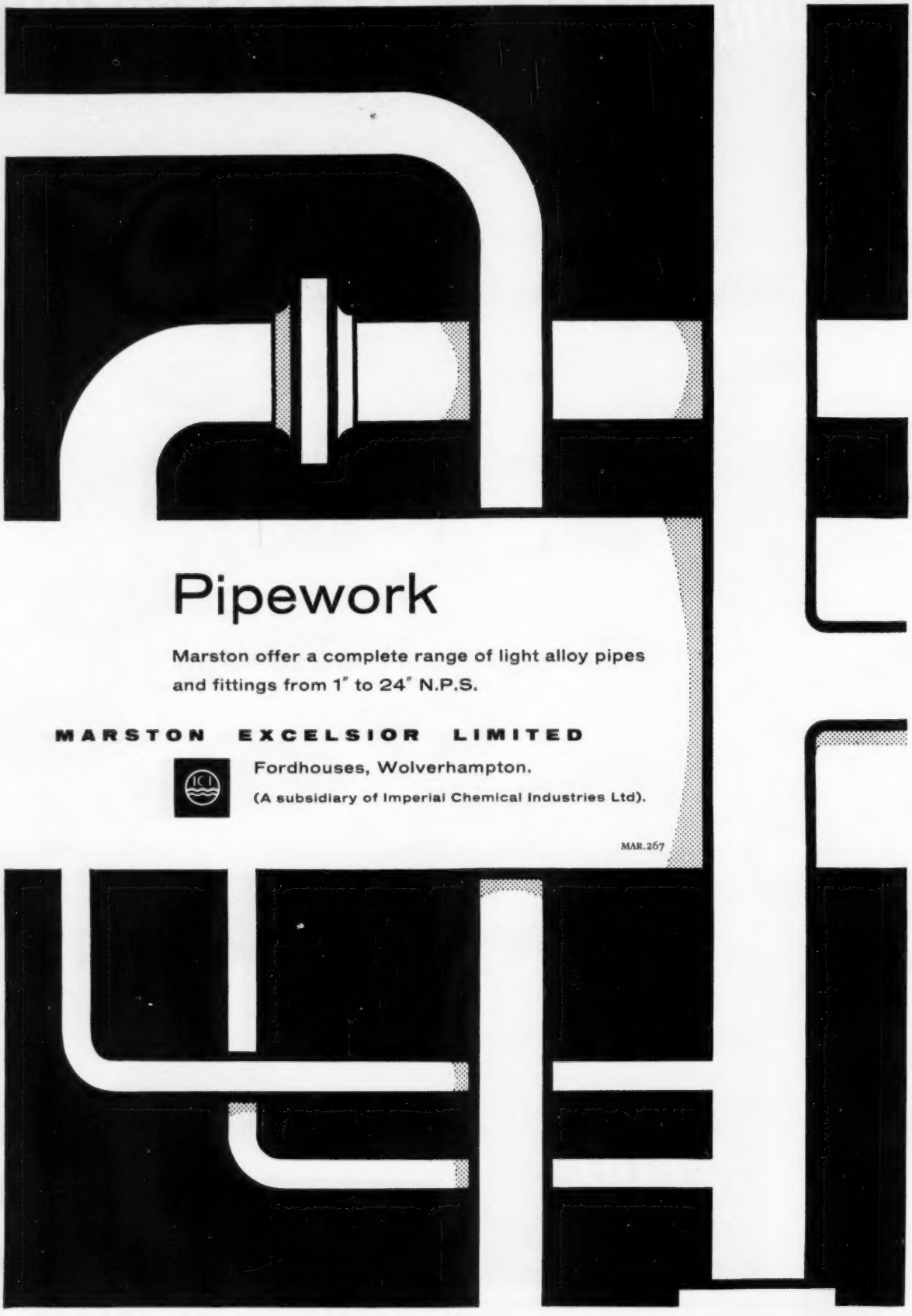
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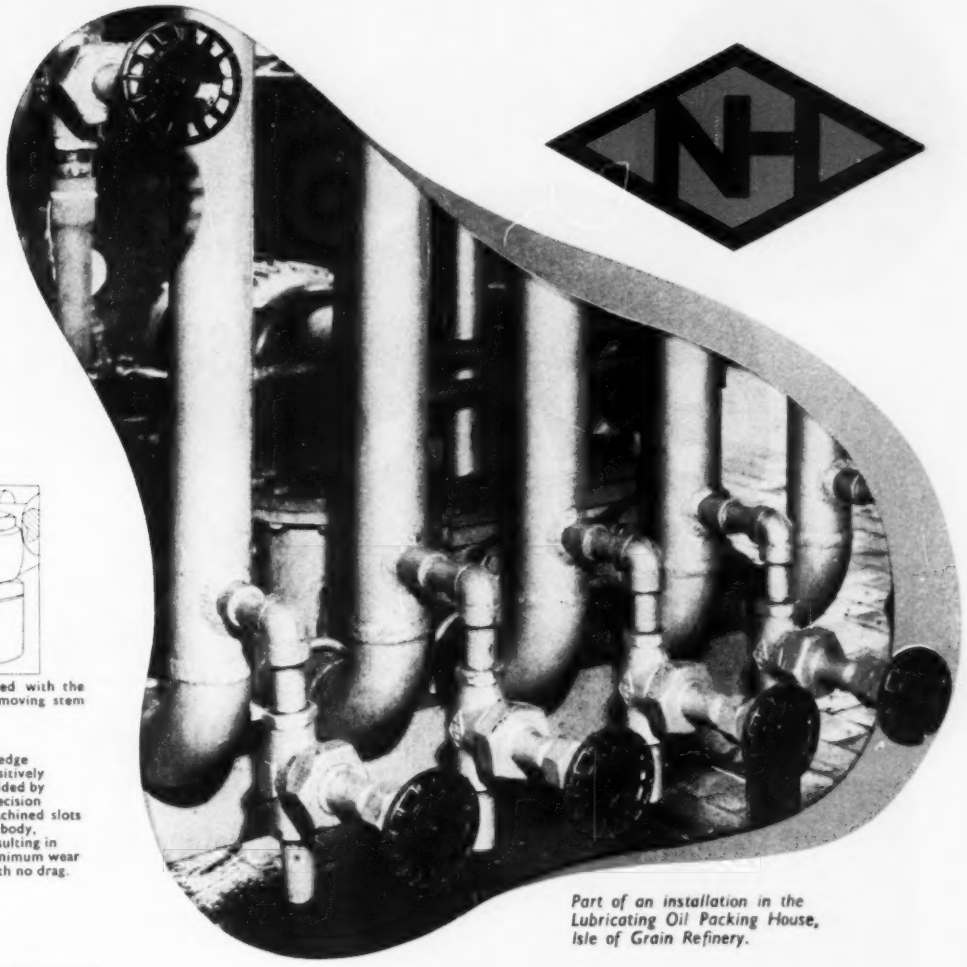
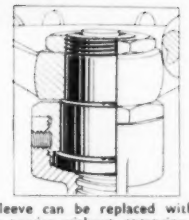


Part of an installation in the Lubricating Oil Packing House, Isle of Grain Refinery.



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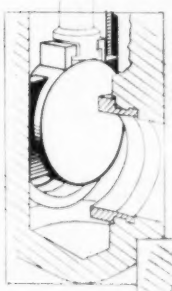

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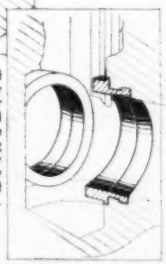

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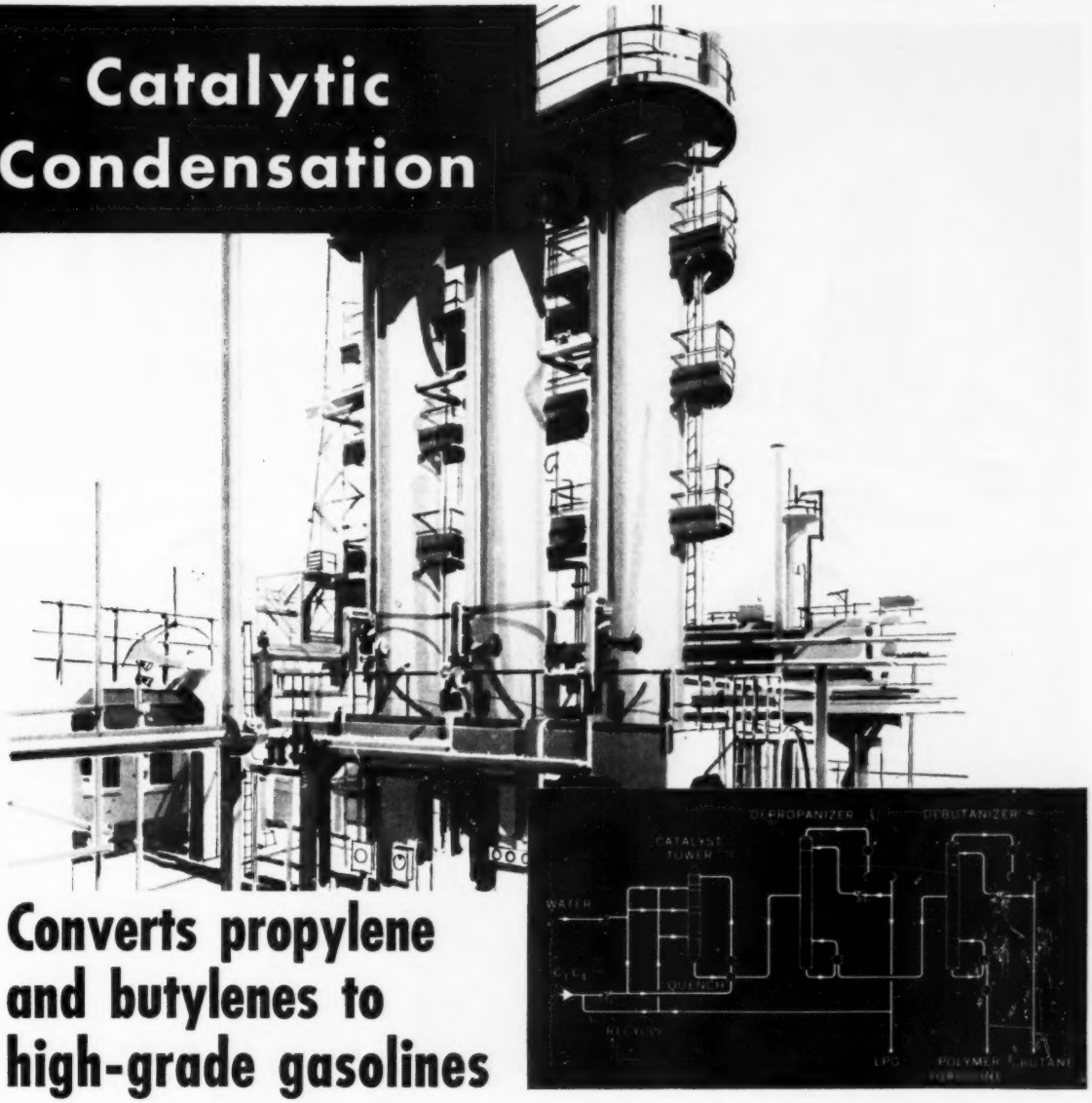

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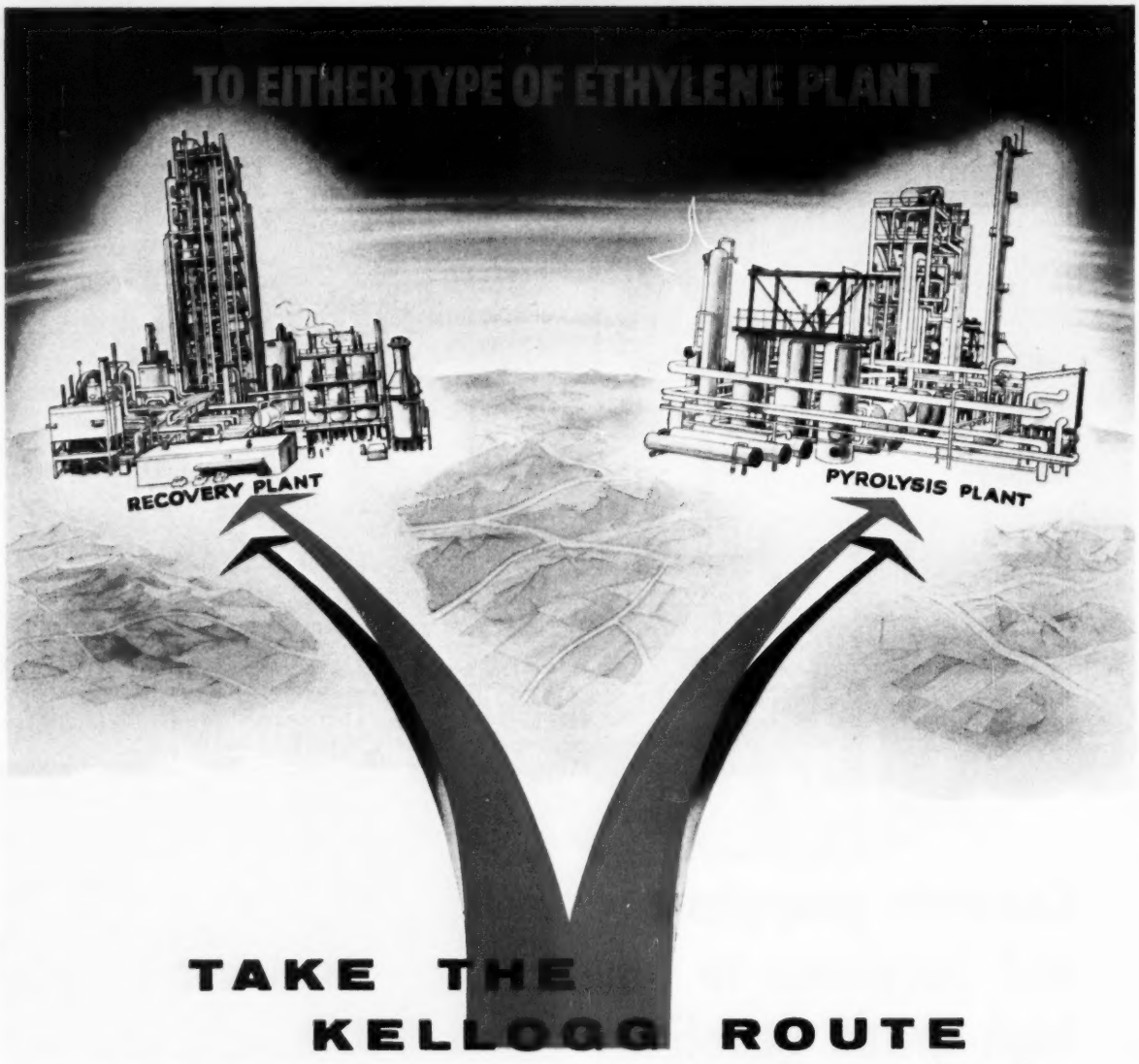

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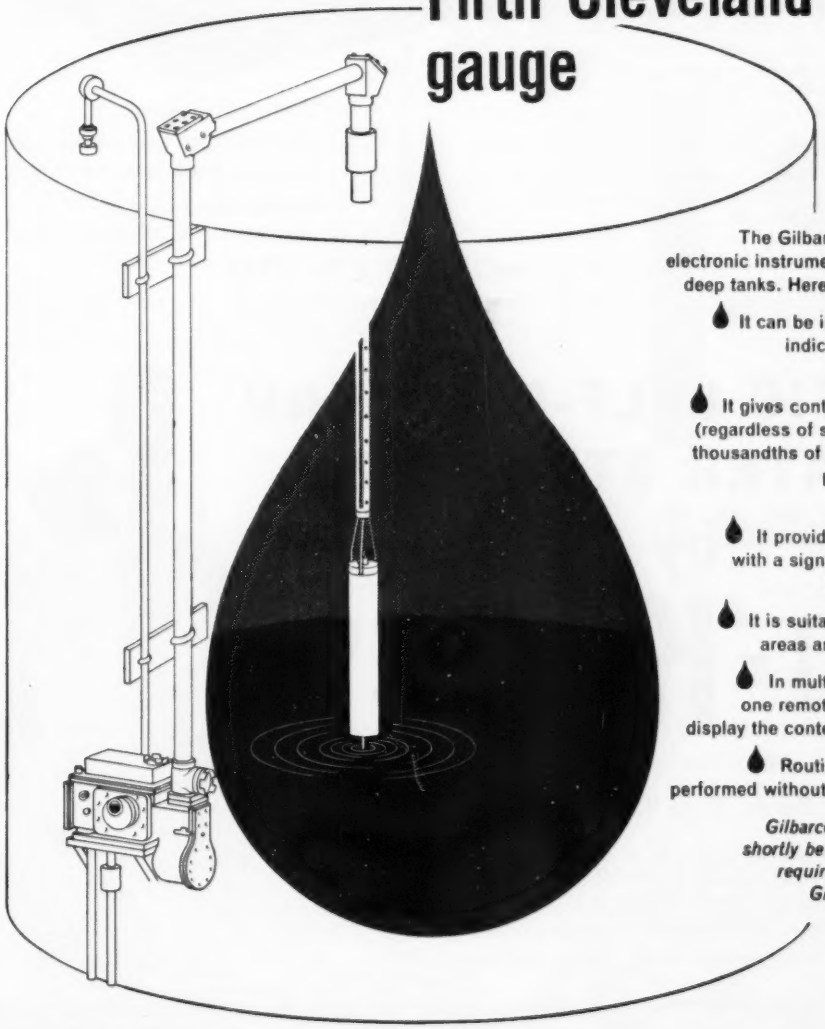

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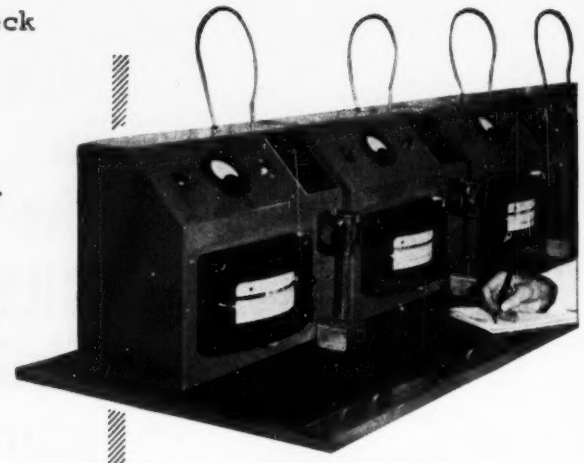

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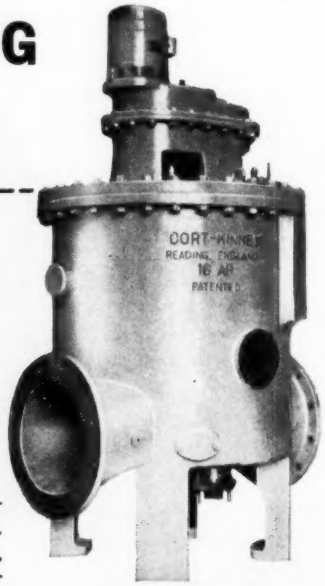

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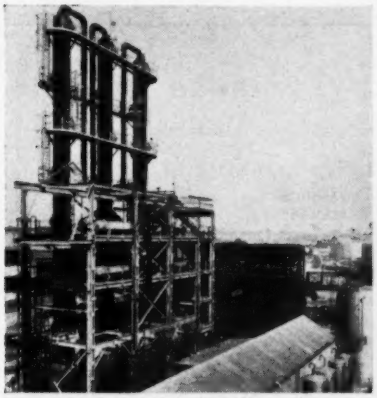

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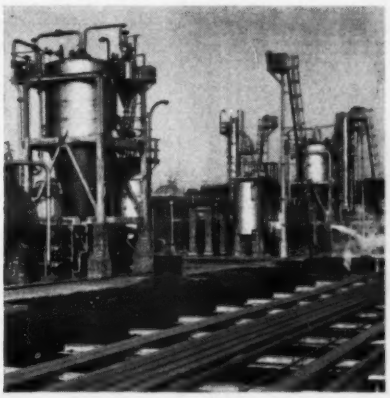

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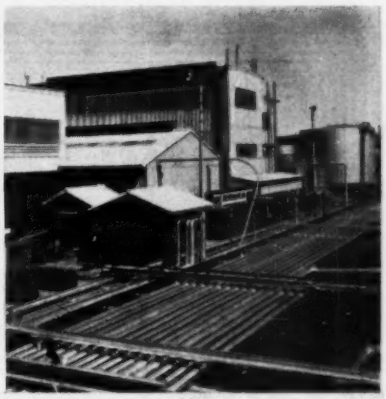

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